Supporting Information Section.

# Synthesis of the Phenoxonium Cation of an α-Tocopherol Model Compound Crystallized With Non-Nucleophilic $[B(C_6F_5)_4]^-$ and $(CB_{11}H_6Br_6)^-$ Anions.

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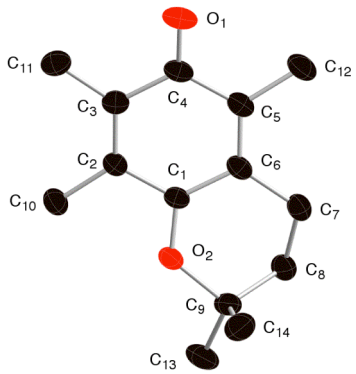
***Table S1***. A selection of crystallographic bond lengths and angles compared with data from molecular orbital calculations using the EDF2/6-31+G* model.[a]

| Bond | Bond Length / Å | | | | |
|---|---|---|---|---|---|
| | $(CH_3)\alpha$-TOH[b] | | $(CH_3)\alpha$-$TO^+$ | | |
| | Crystal | Theoretical | Crystal | | Theoretical |
| | | (EDF2/6–31+G*) | $(CB_{11}H_6Br_6)^-$ | $[B(C_6F_5)]^-$ | (EDF2/6–31+G*) |
| $C_1$–$C_2$ | 1.4055 (14) | 1.404 | 1.468 (7) | 1.455 (4) | 1.454 |
| $C_2$–$C_3$ | 1.3954 (14) | 1.398 | 1.324 (7) | 1.355 (4) | 1.360 |
| $C_3$–$C_4$ | 1.3974 (15) | 1.399 | 1.511 (7) | 1.488 (4) | 1.498 |
| $C_4$–$C_5$ | 1.3929 (15) | 1.397 | 1.488 (7) | 1.491 (4) | 1.501 |
| $C_5$–$C_6$ | 1.4012 (15) | 1.406 | 1.339 (7) | 1.355 (4) | 1.359 |
| $C_6$–$C_1$ | 1.3934 (14) | 1.400 | 1.433 (7) | 1.447 (4) | 1.447 |
| $C_6$–$C_7$ | 1.5132 (14) | 1.511 | 1.520 (7) | 1.505 (4) | 1.504 |
| $C_7$–$C_8$ | 1.5202 (16) | 1.526 | 1.512 (7) | 1.518 (4) | 1.525 |
| $C_8$–$C_9$ | 1.5217 (16) | 1.526 | 1.503 (7) | 1.515 (4) | 1.519 |
| $C_9$–$C_{13}$ | 1.5159 (16) | 1.521 | 1.516 (8) | 1.518 (5) | 1.516 |
| $C_9$–$C_{14}$ | 1.5207 (16) | 1.529 | 1.515 (7) | 1.511 (5) | 1.521 |
| $C_2$–$C_{10}$ | 1.5084 (14) | 1.504 | 1.474 (7) | 1.499 (4) | 1.498 |
| $C_3$–$C_{11}$ | 1.5113 (15) | 1.508 | 1.499 (7) | 1.493 (4) | 1.490 |
| $C_5$–$C_{12}$ | 1.5097 (15) | 1.508 | 1.473 (7) | 1.495 (4) | 1.487 |
| $C_4$–$O_1$ | 1.3950 (13) | 1.377 | 1.219 (6) | 1.214 (3) | 1.217 |
| $C_1$–$O_2$ | 1.3827 (12) | 1.374 | 1.296 (5) | 1.282 (3) | 1.289 |
| $C_9$–$O_2$ | 1.4566 (12) | 1.440 | 1.534 (6) | 1.520 (4) | 1.510 |

| Bond | Bond Angle / ° | | | | |
|---|---|---|---|---|---|
| | $(CH_3)\alpha$-TOH | | $(CH_3)\alpha$-$TO^+$ | | |
| | Crystal | Theoretical | Crystal | | Theoretical |
| | | (EDF2/6–31+G*) | $(CB_{11}H_6Br_6)^-$ | $[B(C_6F_5)]^-$ | (EDF2/6–31+G*) |
| $C_1$–$C_2$–$C_3$ | 118.91 (9) | 119.3 | 117.6 (5) | 117.7 (2) | 118.3 |
| $C_2$–$C_3$–$C_4$ | 119.09 (9) | 118.8 | 119.5 (5) | 118.8 (3) | 119.0 |
| $C_3$–$C_4$–$C_5$ | 122.08 (10) | 122.2 | 119.9 (5) | 120.6 (2) | 120.4 |
| $C_4$–$C_5$–$C_6$ | 118.86 (9) | 119.1 | 119.3 (5) | 119.4 (2) | 119.3 |
| $C_5$–$C_6$–$C_1$ | 119.19 (9) | 118.8 | 118.4 (5) | 117.2 (3) | 118.1 |
| $C_6$–$C_1$–$C_2$ | 121.67 (9) | 121.7 | 124.9 (5) | 125.0 (2) | 124.8 |
| $C_5$–$C_6$–$C_7$ | 120.66 (9) | 121.4 | 122.7 (5) | 123.7 (2) | 123.0 |
| $C_6$–$C_7$–$C_8$ | 111.53 (9) | 112.0 | 110.8 (5) | 110.9 (2) | 111.5 |
| $C_7$–$C_8$–$C_9$ | 111.31 (9) | 111.6 | 112.1 (5) | 111.4 (2) | 112.5 |
| $C_8$–$C_9$–$O_2$ | 107.95 (9) | 108.2 | 107.5 (4) | 107.2 (2) | 107.8 |
| $C_9$–$O_2$–$C_1$ | 117.42 (8) | 118.8 | 122.8 (4) | 123.5 (2) | 124.8 |
| $O_2$–$C_1$–$C_2$ | 115.63 (9) | 115.8 | 113.0 (5) | 113.2 (2) | 113.7 |
| $O_2$–$C_1$–$C_6$ | 122.69 (9) | 122.5 | 122.5 (5) | 121.8 (2) | 121.6 |




[a]The calculated Cartesian coordinates of α-TOH and α-$TO^+$ (which were used to obtain the theoretical bond lengths and bond angles) are given in the Supporting Information section of reference 7c. [b]Values are given for one of the two molecules in the crystallographic asymmetric unit.

16 Feb 2006

## Crystal structure of $(C_{14}H_{19}O_2)^+(CH_6B_{11}Br_6)^-.C_2H_4Cl_2$ –web0601

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### Abstract

The crystal structure of $(C_{14}H_{19}O_2)^+(CH_6B_{11}Br_6)^-.C_2H_4Cl_2$ is reported.

### Comment

The crystallographic asymmetric unit consists of one $(C_{14}H_{19}O_2)^+$ cation, one $(CH_6B_{11}Br_6)^-$ anion and one dichloroethane molecule of solvation. The observation of a peak in a difference electron density map just beyond the H atom on B7 suggests the presence of a low occupancy Br atom site. This could correspond to a small amount of $(CH_5B_{11}Br_7)^-$ impurity (*ca.* 2%). The Br7—B7 distance is shorter than the other Br—B distances, but this could arise from the dodecahedral core of $(CH_5B_{11}Br_7)^-$ being displaced by a small amount with respect to the core of the dominant $(CH_6B_{11}Br_6)^-$ species as a consequence of packing interactions.

The largest peaks in the final difference map are all of near-equal intensity and have no chemical significance.

### Experimental

The compound was prepared by SBL and recrystallized from dichloroethane/acetonitrile. The sample ID is SBL-06.

*Crystal data*

$C_{14}H_{19}O_2^+.CH_{5.98}B_{11}Br_{6.02}^-.C_2H_4Cl_2$
$M_r = 936.19$
Orthorhombic
*Pbca*
$a = 11.5677\,(2)$ Å
$b = 20.2995\,(3)$ Å
$c = 27.9851\,(6)$ Å
$V = 6571.4\,(2)$ Å$^3$
$Z = 8$
$D_x = 1.892$ Mg m$^{-3}$
$D_m$ not measured
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 272638 reflections
$\theta = 3$–$25°$
$\mu = 7.536$ mm$^{-1}$
$T = 200$ K
Plate
Orange
$0.24 \times 0.19 \times 0.06$ mm
Crystal source: local

*Data collection*

Nonius KappaCCD diffractometer
$\varphi$ and $\omega$ scans with CCD
Absorption correction:
by integration *via* Gaussian method (Coppens, 1970) implemented in maXus (2000)
$T_{\min} = 0.342$, $T_{\max} = 0.642$
68824 measured reflections
5825 independent reflections

3223 reflections with
$I > 2.0\sigma(I)$
$R_{\rm int} = 0.076$
$\theta_{\max} = 25.0°$
$h = -13 \rightarrow 13$
$k = -24 \rightarrow 22$
$l = -33 \rightarrow 30$

*Refinement*

Refinement on $F^2$
$R(F) = 0.0321$
$wR(F^2) = 0.0716$
$S = 0.7283$
5806 reflections
410 parameters
H atoms treated by a mixture of independent and constrained refinement

Method= Modified Sheldrick $w$=1/[$\sigma^2(F^2)$ + ( 0.02$P$)$^2$ + 0.00$P$] ,
where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.006837$
$\Delta\rho_{\max} = 1.28$ e Å$^{-3}$
$\Delta\rho_{\min} = -1.13$ e Å$^{-3}$
Extinction correction: none
Scattering factors from International Tables Vol C 4.2.6.8 and 6.1.1.4

Table 1. *Selected geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Br1—B1 | 1.942 (6) | C20—B10 | 1.709 (8) |
| Br2—B2 | 1.942 (6) | C20—B11 | 1.692 (8) |
| Br3—B3 | 1.949 (6) | C51—C52 | 1.481 (8) |
| Br4—B4 | 1.954 (6) | B1—B2 | 1.797 (8) |
| Br5—B5 | 1.953 (6) | B1—B3 | 1.785 (8) |
| Br6—B6 | 1.955 (6) | B1—B4 | 1.788 (9) |
| Cl1—C51 | 1.787 (6) | B1—B5 | 1.795 (8) |
| Cl2—C52 | 1.783 (5) | B1—B6 | 1.792 (8) |
| O1—C4 | 1.219 (6) | B2—B3 | 1.773 (9) |
| O2—C1 | 1.296 (5) | B2—B6 | 1.796 (8) |
| O2—C9 | 1.534 (6) | B2—B7 | 1.753 (9) |
| C1—C2 | 1.468 (7) | B2—B8 | 1.751 (9) |
| C1—C6 | 1.433 (7) | B3—B4 | 1.786 (8) |
| C2—C3 | 1.324 (7) | B3—B8 | 1.763 (8) |
| C2—C10 | 1.474 (7) | B3—B9 | 1.773 (9) |
| C3—C4 | 1.511 (7) | B4—B5 | 1.785 (8) |
| C3—C11 | 1.499 (7) | B4—B9 | 1.770 (8) |
| C4—C5 | 1.488 (7) | B4—B10 | 1.780 (8) |
| C5—C6 | 1.339 (7) | B5—B6 | 1.795 (8) |
| C5—C12 | 1.473 (7) | B5—B10 | 1.779 (8) |
| C6—C7 | 1.520 (7) | B5—B11 | 1.772 (8) |
| C7—C8 | 1.512 (7) | B6—B7 | 1.768 (8) |
| C8—C9 | 1.503 (7) | B6—B11 | 1.780 (8) |
| C9—C13 | 1.516 (8) | B7—B8 | 1.738 (9) |
| C9—C14 | 1.515 (7) | B7—B11 | 1.768 (9) |
| C20—B7 | 1.676 (8) | B8—B9 | 1.782 (9) |
| C20—B8 | 1.690 (8) | B9—B10 | 1.774 (8) |
| C20—B9 | 1.707 (7) | B10—B11 | 1.773 (8) |
| C1—O2—C9 | 122.8 (4) | C3—C4—O1 | 119.4 (5) |
| O2—C1—C2 | 113.0 (5) | C3—C4—C5 | 119.9 (5) |
| O2—C1—C6 | 122.2 (5) | O1—C4—C5 | 120.7 (5) |
| C2—C1—C6 | 124.9 (5) | C4—C5—C6 | 119.3 (5) |
| C1—C2—C3 | 117.6 (5) | C4—C5—C12 | 116.6 (5) |
| C1—C2—C10 | 118.0 (5) | C6—C5—C12 | 124.1 (5) |
| C3—C2—C10 | 124.5 (5) | C1—C6—C5 | 118.4 (5) |
| C2—C3—C4 | 119.5 (5) | C1—C6—C7 | 118.9 (5) |
| C2—C3—C11 | 125.2 (5) | C5—C6—C7 | 122.7 (5) |
| C4—C3—C11 | 115.3 (5) | C6—C7—C8 | 110.8 (5) |

| | | | |
|---|---|---|---|
| C7—C8—C9 | 112.1 (4) | B3—B2—B6 | 107.9 (4) |
| O2—C9—C8 | 107.5 (4) | B1—B2—B7 | 107.2 (4) |
| O2—C9—C13 | 101.6 (5) | Br2—B2—B7 | 122.9 (4) |
| C8—C9—C13 | 113.8 (5) | B3—B2—B7 | 107.2 (5) |
| O2—C9—C14 | 104.3 (4) | B6—B2—B7 | 59.7 (3) |
| C8—C9—C14 | 115.0 (5) | B1—B2—B8 | 107.9 (4) |
| C13—C9—C14 | 113.0 (5) | Br2—B2—B8 | 122.5 (4) |
| B7—C20—B8 | 62.2 (4) | B3—B2—B8 | 60.0 (3) |
| B7—C20—B9 | 114.7 (4) | B6—B2—B8 | 107.7 (4) |
| B8—C20—B9 | 63.3 (3) | B7—B2—B8 | 59.5 (4) |
| B7—C20—B10 | 115.2 (4) | B1—B3—B2 | 60.7 (3) |
| B8—C20—B10 | 115.2 (4) | B1—B3—Br3 | 120.9 (4) |
| B9—C20—B10 | 62.6 (3) | B2—B3—Br3 | 121.8 (4) |
| B7—C20—B11 | 63.3 (4) | B1—B3—B4 | 60.1 (3) |
| B8—C20—B11 | 115.1 (4) | B2—B3—B4 | 108.6 (4) |
| B9—C20—B11 | 114.8 (4) | Br3—B3—B4 | 121.0 (4) |
| B10—C20—B11 | 62.8 (3) | B1—B3—B8 | 107.8 (4) |
| Cl1—C51—C52 | 111.7 (4) | B2—B3—B8 | 59.4 (3) |
| C51—C52—Cl2 | 113.4 (4) | Br3—B3—B8 | 122.8 (4) |
| Br1—B1—B2 | 121.9 (4) | B4—B3—B8 | 107.9 (4) |
| Br1—B1—B3 | 121.7 (4) | B1—B3—B9 | 108.0 (4) |
| B2—B1—B3 | 59.3 (3) | B2—B3—B9 | 108.3 (4) |
| Br1—B1—B4 | 121.8 (4) | Br3—B3—B9 | 121.8 (4) |
| B2—B1—B4 | 107.5 (4) | B4—B3—B9 | 59.6 (3) |
| B3—B1—B4 | 60.0 (3) | B8—B3—B9 | 60.5 (3) |
| Br1—B1—B5 | 122.2 (4) | B1—B4—B3 | 59.9 (3) |
| B2—B1—B5 | 107.8 (4) | B1—B4—Br4 | 122.9 (4) |
| B3—B1—B5 | 107.6 (4) | B3—B4—Br4 | 121.9 (4) |
| B4—B1—B5 | 59.8 (3) | B1—B4—B5 | 60.3 (3) |
| Br1—B1—B6 | 122.1 (4) | B3—B4—B5 | 108.0 (4) |
| B2—B1—B6 | 60.1 (3) | Br4—B4—B5 | 122.5 (4) |
| B3—B1—B6 | 107.5 (4) | B1—B4—B9 | 108.0 (4) |
| B4—B1—B6 | 107.8 (4) | B3—B4—B9 | 59.8 (3) |
| B5—B1—B6 | 60.1 (3) | Br4—B4—B9 | 120.4 (4) |
| B1—B2—Br2 | 121.2 (4) | B5—B4—B9 | 107.9 (4) |
| B1—B2—B3 | 60.0 (3) | B1—B4—B10 | 108.2 (4) |
| Br2—B2—B3 | 121.8 (4) | B3—B4—B10 | 107.7 (4) |
| B1—B2—B6 | 59.8 (3) | Br4—B4—B10 | 120.9 (4) |
| Br2—B2—B6 | 121.4 (4) | B5—B4—B10 | 59.9 (3) |

| | | | |
|---|---|---|---|
| B9—B4—B10 | 60.0 (3) | B2—B7—B11 | 109.6 (4) |
| B1—B5—B4 | 59.9 (3) | B8—B7—B11 | 108.9 (5) |
| B1—B5—Br5 | 122.3 (4) | C20—B8—B3 | 104.9 (4) |
| B4—B5—Br5 | 122.0 (4) | C20—B8—B2 | 105.4 (4) |
| B1—B5—B6 | 59.9 (3) | B3—B8—B2 | 60.6 (4) |
| B4—B5—B6 | 107.8 (4) | C20—B8—B7 | 58.5 (3) |
| Br5—B5—B6 | 121.9 (4) | B3—B8—B7 | 108.3 (4) |
| B1—B5—B10 | 107.9 (4) | B2—B8—B7 | 60.3 (4) |
| B4—B5—B10 | 59.9 (3) | C20—B8—B9 | 58.8 (3) |
| Br5—B5—B10 | 121.4 (4) | B3—B8—B9 | 60.0 (3) |
| B6—B5—B10 | 108.0 (4) | B2—B8—B9 | 108.9 (4) |
| B1—B5—B11 | 107.5 (4) | B7—B8—B9 | 108.0 (5) |
| B4—B5—B11 | 107.6 (4) | C20—B9—B8 | 57.9 (3) |
| Br5—B5—B11 | 121.8 (4) | C20—B9—B3 | 103.8 (4) |
| B6—B5—B11 | 59.9 (3) | B8—B9—B3 | 59.5 (4) |
| B10—B5—B11 | 59.9 (3) | C20—B9—B4 | 104.6 (4) |
| B2—B6—B5 | 107.8 (4) | B8—B9—B4 | 107.8 (4) |
| B2—B6—B1 | 60.1 (3) | B3—B9—B4 | 60.6 (3) |
| B5—B6—B1 | 60.1 (3) | C20—B9—B10 | 58.8 (3) |
| B2—B6—Br6 | 123.2 (4) | B8—B9—B10 | 107.7 (4) |
| B5—B6—Br6 | 121.5 (4) | B3—B9—B10 | 108.6 (4) |
| B1—B6—Br6 | 122.9 (4) | B4—B9—B10 | 60.3 (3) |
| B2—B6—B7 | 58.9 (3) | C20—B10—B4 | 104.0 (4) |
| B5—B6—B7 | 106.9 (4) | C20—B10—B5 | 104.0 (4) |
| B1—B6—B7 | 106.7 (4) | B4—B10—B5 | 60.2 (3) |
| Br6—B6—B7 | 122.5 (4) | C20—B10—B9 | 58.7 (3) |
| B2—B6—B11 | 107.1 (4) | B4—B10—B9 | 59.7 (3) |
| B5—B6—B11 | 59.4 (3) | B5—B10—B9 | 108.0 (4) |
| B1—B6—B11 | 107.3 (4) | C20—B10—B11 | 58.1 (3) |
| Br6—B6—B11 | 120.9 (4) | B4—B10—B11 | 107.7 (4) |
| B7—B6—B11 | 59.8 (3) | B5—B10—B11 | 59.9 (3) |
| C20—B7—B6 | 105.8 (4) | B9—B10—B11 | 107.7 (5) |
| C20—B7—B2 | 105.9 (4) | C20—B11—B6 | 104.6 (4) |
| B6—B7—B2 | 61.4 (3) | C20—B11—B10 | 59.1 (3) |
| C20—B7—B8 | 59.3 (3) | B6—B11—B10 | 108.9 (4) |
| B6—B7—B8 | 109.6 (4) | C20—B11—B5 | 105.0 (4) |
| B2—B7—B8 | 60.2 (4) | B6—B11—B5 | 60.7 (3) |
| C20—B7—B11 | 58.8 (3) | B10—B11—B5 | 60.2 (3) |
| B6—B7—B11 | 60.5 (3) | C20—B11—B7 | 57.9 (3) |

| | | | |
|---|---|---|---|
| B6—B11—B7 | 59.8 (3) | B5—B11—B7 | 107.9 (4) |
| B10—B11—B7 | 107.7 (4) | | |

H atoms were included at calculated positions with methyl H atoms oriented to best-match peaks in difference electron density maps. Restraints were imposed on distances and angles involving H atoms of the cation as they were refined positionally. Other H atoms ride on the atom to which they are attached.

Data collection: COLLECT (Nonius BV, 1997). Cell refinement: Denzo/Scalepack . Data reduction: Denzo/Scalepack (Otwinowski & Minor, 1996). Program(s) used to solve structure: *SIR*92 (Altomare *et al.* 1994). Program(s) used to refine structure: *CRYSTALS* (Watkin *et al.*2003). Molecular graphics: *ORTEP*–II (Johnson 1976) in teXsan (MSC, 1992–1997) . Software used to prepare material for publication: *CRYSTALS* .

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## Supplementary data

Table *S*1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$$U_{\mathrm{eq}} = (1/3)\Sigma_i\Sigma_j U^{ij} a^i a^j \mathbf{a}_i.\mathbf{a}_j.$$

| | Occupancy | $x$ | $y$ | $z$ | $U_{\mathrm{eq}}$ |
|---|---|---|---|---|---|
| Br1 | 1.0000 | 0.60711 (5) | 0.33738 (3) | 0.07643 (2) | 0.0481 |
| Br2 | 1.0000 | 0.29738 (5) | 0.39726 (3) | 0.05529 (2) | 0.0621 |
| Br3 | 1.0000 | 0.35642 (5) | 0.25449 (3) | 0.14091 (2) | 0.0560 |
| Br4 | 1.0000 | 0.63405 (5) | 0.29075 (3) | 0.21013 (2) | 0.0491 |
| Br5 | 1.0000 | 0.75277 (5) | 0.45788 (3) | 0.16527 (2) | 0.0429 |
| Br6 | 1.0000 | 0.54366 (5) | 0.52517 (3) | 0.07012 (2) | 0.0492 |
| Br7 | 0.0194 (16) | 0.266 (2) | 0.5476 (13) | 0.1320 (11) | 0.0449 |
| Cl1 | 1.0000 | 0.81035 (15) | 0.34192 (9) | −0.02580 (7) | 0.0842 |
| Cl2 | 1.0000 | 0.98812 (13) | 0.43320 (7) | 0.03798 (6) | 0.0585 |
| O1 | 1.0000 | 0.5768 (3) | 0.08948 (18) | 0.21772 (14) | 0.0528 |
| O2 | 1.0000 | 0.8609 (3) | 0.20860 (17) | 0.09978 (13) | 0.0441 |
| C1 | 1.0000 | 0.7998 (4) | 0.1751 (2) | 0.13004 (19) | 0.0344 |
| C2 | 1.0000 | 0.6938 (4) | 0.1479 (2) | 0.10895 (19) | 0.0335 |
| C3 | 1.0000 | 0.6209 (4) | 0.1174 (2) | 0.1379 (2) | 0.0406 |
| C4 | 1.0000 | 0.6488 (5) | 0.1120 (2) | 0.1905 (2) | 0.0356 |
| C5 | 1.0000 | 0.7627 (4) | 0.1353 (2) | 0.20859 (19) | 0.0343 |
| C6 | 1.0000 | 0.8352 (4) | 0.1660 (2) | 0.17865 (19) | 0.0333 |
| C7 | 1.0000 | 0.9526 (4) | 0.1917 (2) | 0.1943 (2) | 0.0442 |
| C8 | 1.0000 | 1.0311 (4) | 0.2015 (3) | 0.1516 (2) | 0.0531 |
| C9 | 1.0000 | 0.9749 (4) | 0.2424 (3) | 0.1134 (2) | 0.0500 |
| C10 | 1.0000 | 0.6748 (5) | 0.1575 (3) | 0.0573 (2) | 0.0542 |
| C11 | 1.0000 | 0.5088 (5) | 0.0865 (3) | 0.1229 (2) | 0.0579 |
| C12 | 1.0000 | 0.7865 (5) | 0.1242 (3) | 0.2597 (2) | 0.0530 |
| C13 | 1.0000 | 1.0392 (5) | 0.2417 (3) | 0.0662 (2) | 0.0787 |
| C14 | 1.0000 | 0.9392 (5) | 0.3110 (3) | 0.1288 (2) | 0.0584 |
| C20 | 1.0000 | 0.3738 (5) | 0.4579 (2) | 0.20776 (19) | 0.0424 |
| C51 | 1.0000 | 0.7939 (5) | 0.4287 (3) | −0.0176 (2) | 0.0588 |
| C52 | 1.0000 | 0.8383 (4) | 0.4504 (3) | 0.0295 (2) | 0.0536 |
| B1 | 1.0000 | 0.5202 (5) | 0.3829 (3) | 0.1258 (2) | 0.0332 |
| B2 | 1.0000 | 0.3745 (5) | 0.4107 (3) | 0.1161 (2) | 0.0415 |
| B3 | 1.0000 | 0.4020 (5) | 0.3446 (3) | 0.1559 (2) | 0.0392 |
| B4 | 1.0000 | 0.5331 (5) | 0.3613 (3) | 0.1875 (2) | 0.0359 |
| B5 | 1.0000 | 0.5874 (5) | 0.4386 (3) | 0.1671 (2) | 0.0345 |
| B6 | 1.0000 | 0.4898 (5) | 0.4693 (3) | 0.1225 (2) | 0.0358 |
| B7 | 1.0000 | 0.3550 (5) | 0.4816 (3) | 0.1509 (2) | 0.0453 |
| B8 | 1.0000 | 0.3014 (5) | 0.4065 (3) | 0.1708 (2) | 0.0437 |
| B9 | 1.0000 | 0.3978 (5) | 0.3755 (3) | 0.2151 (2) | 0.0437 |
| B10 | 1.0000 | 0.5110 (5) | 0.4337 (3) | 0.2221 (2) | 0.0369 |
| B11 | 1.0000 | 0.4841 (5) | 0.4997 (3) | 0.1821 (2) | 0.0414 |
| H7 | 0.9810 | 0.3010 (5) | 0.5178 (3) | 0.1415 (2) | 0.0531 |
| H8 | 1.0000 | 0.2164 (5) | 0.3983 (3) | 0.1734 (2) | 0.0525 |
| H9 | 1.0000 | 0.3686 (5) | 0.3498 (3) | 0.2431 (2) | 0.0478 |
| H10 | 1.0000 | 0.5476 (5) | 0.4419 (3) | 0.2540 (2) | 0.0470 |
| H11 | 1.0000 | 0.5051 (5) | 0.5459 (3) | 0.1908 (2) | 0.0520 |
| H20 | 1.0000 | 0.3281 (5) | 0.4813 (2) | 0.23288 (19) | 0.0542 |
| H71 | 1.0000 | 0.985 (2) | 0.1608 (12) | 0.2166 (9) | 0.0582 |
| H72 | 1.0000 | 0.943 (2) | 0.2324 (11) | 0.2105 (9) | 0.0582 |
| H81 | 1.0000 | 1.100 (2) | 0.2229 (13) | 0.1623 (9) | 0.0630 |
| H82 | 1.0000 | 1.052 (2) | 0.1593 (11) | 0.1378 (9) | 0.0630 |
| H101 | 1.0000 | 0.607 (2) | 0.1367 (16) | 0.0478 (9) | 0.0671 |
| H102 | 1.0000 | 0.738 (2) | 0.1404 (17) | 0.0397 (9) | 0.0671 |
| H103 | 1.0000 | 0.668 (3) | 0.2021 (10) | 0.0503 (9) | 0.0671 |
| H111 | 1.0000 | 0.458 (2) | 0.0848 (17) | 0.1488 (8) | 0.0695 |
| H112 | 1.0000 | 0.521 (2) | 0.0434 (11) | 0.1117 (13) | 0.0695 |
| H113 | 1.0000 | 0.477 (2) | 0.1113 (14) | 0.0977 (10) | 0.0695 |
| H121 | 1.0000 | 0.7180 (19) | 0.1121 (16) | 0.2764 (9) | 0.0618 |
| H122 | 1.0000 | 0.840 (3) | 0.0887 (13) | 0.2627 (9) | 0.0618 |

| | | | | | |
|---|---|---|---|---|---|
| H123 | 1.0000 | 0.818 (3) | 0.1620 (12) | 0.2742 (9) | 0.0618 |
| H131 | 1.0000 | 1.111 (2) | 0.2637 (18) | 0.0708 (10) | 0.0946 |
| H132 | 1.0000 | 1.052 (3) | 0.1962 (11) | 0.0566 (11) | 0.0946 |
| H133 | 1.0000 | 0.994 (3) | 0.2641 (18) | 0.0419 (9) | 0.0946 |
| H141 | 1.0000 | 1.0051 (19) | 0.3372 (12) | 0.1337 (13) | 0.0707 |
| H142 | 1.0000 | 0.896 (3) | 0.3096 (13) | 0.1576 (10) | 0.0707 |
| H143 | 1.0000 | 0.893 (3) | 0.3316 (13) | 0.1041 (10) | 0.0707 |
| H511 | 1.0000 | 0.8370 (5) | 0.4522 (3) | −0.0435 (2) | 0.0717 |
| H512 | 1.0000 | 0.7099 (5) | 0.4401 (3) | −0.0196 (2) | 0.0717 |
| H521 | 1.0000 | 0.8270 (4) | 0.4991 (3) | 0.0319 (2) | 0.0612 |
| H522 | 1.0000 | 0.7932 (4) | 0.4279 (3) | 0.0552 (2) | 0.0612 |

Table *S*2. *Anisotropic displacement parameters* (Å$^2$)

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|
| Br1 | 0.0473 (3) | 0.0452 (3) | 0.0518 (4) | −0.0031 (3) | 0.0139 (3) | −0.0153 (3) |
| Br2 | 0.0551 (4) | 0.0758 (5) | 0.0555 (4) | −0.0056 (4) | −0.0198 (3) | −0.0021 (4) |
| Br3 | 0.0482 (3) | 0.0395 (3) | 0.0804 (5) | −0.0146 (3) | 0.0027 (3) | −0.0058 (3) |
| Br4 | 0.0431 (3) | 0.0362 (3) | 0.0681 (4) | 0.0033 (3) | −0.0036 (3) | 0.0113 (3) |
| Br5 | 0.0375 (3) | 0.0398 (3) | 0.0514 (4) | −0.0091 (3) | −0.0047 (3) | −0.0002 (3) |
| Br6 | 0.0565 (4) | 0.0462 (3) | 0.0450 (4) | −0.0019 (3) | 0.0031 (3) | 0.0124 (3) |
| Br7 | 0.047 (14) | 0.034 (13) | 0.054 (15) | 0.017 (11) | −0.008 (12) | 0.016 (12) |
| Cl1 | 0.0753 (12) | 0.0784 (12) | 0.0989 (15) | −0.0216 (10) | 0.0263 (11) | −0.0382 (11) |
| Cl2 | 0.0517 (9) | 0.0527 (9) | 0.0712 (12) | −0.0050 (8) | 0.0051 (8) | 0.0110 (9) |
| O1 | 0.047 (2) | 0.050 (2) | 0.061 (3) | 0.001 (2) | 0.018 (2) | 0.002 (2) |
| O2 | 0.041 (2) | 0.044 (2) | 0.047 (3) | 0.0008 (19) | 0.002 (2) | 0.006 (2) |
| C1 | 0.037 (3) | 0.025 (3) | 0.041 (4) | 0.010 (3) | 0.010 (3) | 0.006 (3) |
| C2 | 0.037 (3) | 0.027 (3) | 0.036 (4) | 0.005 (3) | −0.001 (3) | −0.004 (3) |
| C3 | 0.039 (3) | 0.034 (3) | 0.049 (4) | 0.004 (3) | −0.005 (3) | −0.015 (3) |
| C4 | 0.041 (3) | 0.024 (3) | 0.041 (4) | 0.003 (3) | 0.006 (3) | −0.003 (3) |
| C5 | 0.037 (3) | 0.032 (3) | 0.034 (3) | 0.008 (3) | −0.006 (3) | −0.003 (3) |
| C6 | 0.032 (3) | 0.027 (3) | 0.041 (4) | 0.003 (2) | −0.006 (3) | −0.001 (3) |
| C7 | 0.041 (3) | 0.031 (3) | 0.061 (4) | −0.001 (3) | −0.008 (3) | −0.002 (3) |
| C8 | 0.031 (3) | 0.043 (3) | 0.085 (5) | −0.001 (3) | −0.002 (3) | 0.004 (4) |
| C9 | 0.037 (3) | 0.046 (4) | 0.066 (5) | −0.005 (3) | 0.009 (3) | 0.016 (3) |
| C10 | 0.066 (4) | 0.053 (4) | 0.043 (4) | −0.005 (3) | −0.012 (3) | −0.002 (3) |
| C11 | 0.037 (3) | 0.058 (4) | 0.079 (5) | −0.001 (3) | −0.007 (3) | −0.009 (4) |
| C12 | 0.051 (4) | 0.059 (4) | 0.049 (5) | −0.003 (3) | 0.001 (3) | 0.006 (3) |
| C13 | 0.061 (4) | 0.082 (5) | 0.093 (6) | −0.004 (4) | 0.028 (4) | 0.025 (5) |
| C14 | 0.054 (4) | 0.035 (3) | 0.086 (5) | −0.006 (3) | −0.001 (4) | 0.013 (3) |
| C20 | 0.049 (3) | 0.040 (3) | 0.039 (4) | 0.008 (3) | 0.007 (3) | 0.002 (3) |
| C51 | 0.048 (4) | 0.066 (4) | 0.062 (5) | −0.004 (3) | 0.008 (4) | −0.002 (4) |
| C52 | 0.045 (3) | 0.050 (4) | 0.066 (5) | −0.006 (3) | 0.017 (3) | 0.002 (3) |
| B1 | 0.029 (3) | 0.032 (3) | 0.039 (4) | 0.000 (3) | 0.005 (3) | −0.006 (3) |
| B2 | 0.034 (4) | 0.046 (4) | 0.045 (4) | −0.002 (3) | −0.010 (3) | 0.000 (3) |
| B3 | 0.030 (3) | 0.039 (4) | 0.048 (4) | 0.002 (3) | 0.001 (3) | −0.002 (3) |
| B4 | 0.034 (3) | 0.029 (3) | 0.045 (4) | 0.003 (3) | −0.001 (3) | 0.003 (3) |
| B5 | 0.041 (3) | 0.029 (3) | 0.034 (4) | −0.003 (3) | 0.000 (3) | 0.000 (3) |
| B6 | 0.039 (4) | 0.039 (4) | 0.030 (4) | −0.001 (3) | 0.001 (3) | 0.002 (3) |
| B7 | 0.040 (4) | 0.039 (4) | 0.057 (5) | 0.014 (3) | 0.004 (3) | 0.006 (3) |
| B8 | 0.034 (3) | 0.046 (4) | 0.051 (5) | 0.002 (3) | 0.006 (3) | 0.006 (4) |
| B9 | 0.041 (4) | 0.042 (4) | 0.048 (5) | 0.004 (3) | 0.021 (3) | 0.009 (3) |
| B10 | 0.048 (4) | 0.032 (3) | 0.031 (4) | −0.002 (3) | 0.001 (3) | 0.000 (3) |
| B11 | 0.048 (4) | 0.034 (3) | 0.043 (4) | 0.006 (3) | 0.003 (3) | −0.009 (3) |

Table *S*3. *Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Br1—B1 | 1.942 (6) | C14—H142 | 0.947 (19) |
| Br2—B2 | 1.942 (6) | C14—H143 | 0.970 (19) |
| Br3—B3 | 1.949 (6) | C20—B7 | 1.676 (8) |
| Br4—B4 | 1.954 (6) | C20—B8 | 1.690 (8) |
| Br5—B5 | 1.953 (6) | C20—B9 | 1.707 (7) |
| Br6—B6 | 1.955 (6) | C20—B10 | 1.709 (8) |
| Br7—B7 | 1.77 (2) | C20—B11 | 1.692 (8) |
| Cl1—C51 | 1.787 (6) | C20—H20 | 1.000 |
| Cl2—C52 | 1.783 (5) | C51—C52 | 1.481 (8) |
| O1—C4 | 1.219 (6) | C51—H511 | 1.000 |
| O2—C1 | 1.296 (5) | C51—H512 | 1.000 |
| O2—C9 | 1.534 (6) | C52—H521 | 1.000 |
| C1—C2 | 1.468 (7) | C52—H522 | 1.000 |
| C1—C6 | 1.433 (7) | B1—B2 | 1.797 (8) |
| C2—C3 | 1.324 (7) | B1—B3 | 1.785 (8) |
| C2—C10 | 1.474 (7) | B1—B4 | 1.788 (9) |
| C3—C4 | 1.511 (7) | B1—B5 | 1.795 (8) |
| C3—C11 | 1.499 (7) | B1—B6 | 1.792 (8) |
| C4—C5 | 1.488 (7) | B2—B3 | 1.773 (9) |
| C5—C6 | 1.339 (7) | B2—B6 | 1.796 (8) |
| C5—C12 | 1.473 (7) | B2—B7 | 1.753 (9) |
| C6—C7 | 1.520 (7) | B2—B8 | 1.751 (9) |
| C7—C8 | 1.512 (7) | B3—B4 | 1.786 (8) |
| C7—H71 | 0.963 (19) | B3—B8 | 1.763 (8) |
| C7—H72 | 0.949 (18) | B3—B9 | 1.773 (9) |
| C8—C9 | 1.503 (7) | B4—B5 | 1.785 (8) |
| C8—H81 | 0.960 (19) | B4—B9 | 1.770 (8) |
| C8—H82 | 0.973 (19) | B4—B10 | 1.780 (8) |
| C9—C13 | 1.516 (8) | B5—B6 | 1.795 (8) |
| C9—C14 | 1.515 (7) | B5—B10 | 1.779 (8) |
| C10—H101 | 0.930 (19) | B5—B11 | 1.772 (8) |
| C10—H102 | 0.947 (19) | B6—B7 | 1.768 (8) |
| C10—H103 | 0.930 (19) | B6—B11 | 1.780 (8) |
| C11—H111 | 0.934 (19) | B7—B8 | 1.738 (9) |
| C11—H112 | 0.939 (19) | B7—B11 | 1.768 (9) |
| C11—H113 | 0.942 (19) | B7—H7 | 1.000 |
| C12—H121 | 0.952 (19) | B8—B9 | 1.782 (9) |
| C12—H122 | 0.950 (19) | B8—H8 | 1.000 |
| C12—H123 | 0.945 (19) | B9—B10 | 1.774 (8) |
| C13—H131 | 0.950 (19) | B9—H9 | 1.000 |
| C13—H132 | 0.974 (19) | B10—B11 | 1.773 (8) |
| C13—H133 | 0.968 (19) | B10—H10 | 1.000 |
| C14—H141 | 0.939 (19) | B11—H11 | 1.000 |
| C1—O2—C9 | 122.8 (4) | H71—C7—H72 | 107.6 (17) |
| O2—C1—C2 | 113.0 (5) | C7—C8—C9 | 112.1 (4) |
| O2—C1—C6 | 122.2 (5) | C7—C8—H81 | 108.3 (15) |
| C2—C1—C6 | 124.9 (5) | C9—C8—H81 | 109.5 (15) |
| C1—C2—C3 | 117.6 (5) | C7—C8—H82 | 110.4 (15) |
| C1—C2—C10 | 118.0 (5) | C9—C8—H82 | 108.1 (15) |
| C3—C2—C10 | 124.5 (5) | H81—C8—H82 | 108.3 (17) |
| C2—C3—C4 | 119.5 (5) | O2—C9—C8 | 107.5 (4) |
| C2—C3—C11 | 125.2 (5) | O2—C9—C13 | 101.6 (5) |
| C4—C3—C11 | 115.3 (5) | C8—C9—C13 | 113.8 (5) |
| C3—C4—O1 | 119.4 (5) | O2—C9—C14 | 104.3 (4) |
| C3—C4—C5 | 119.9 (5) | C8—C9—C14 | 115.0 (5) |
| O1—C4—C5 | 120.7 (5) | C13—C9—C14 | 113.0 (5) |
| C4—C5—C6 | 119.3 (5) | C2—C10—H101 | 110.4 (16) |
| C4—C5—C12 | 116.6 (5) | C2—C10—H102 | 110.3 (16) |
| C6—C5—C12 | 124.1 (5) | H101—C10—H102 | 109.5 (17) |
| C1—C6—C5 | 118.4 (5) | C2—C10—H103 | 110.4 (16) |
| C1—C6—C7 | 118.9 (5) | H101—C10—H103 | 107.9 (17) |
| C5—C6—C7 | 122.7 (5) | H102—C10—H103 | 108.4 (17) |
| C6—C7—C8 | 110.8 (5) | C3—C11—H111 | 110.0 (16) |
| C6—C7—H71 | 108.3 (15) | C3—C11—H112 | 110.8 (16) |
| C8—C7—H71 | 111.2 (15) | H111—C11—H112 | 108.4 (17) |
| C6—C7—H72 | 109.7 (15) | C3—C11—H113 | 108.9 (16) |
| C8—C7—H72 | 109.2 (15) | H111—C11—H113 | 110.9 (17) |

| | |
|---|---|
| H112—C11—H113 | 107.8 (17) |
| C5—C12—H121 | 111.1 (16) |
| C5—C12—H122 | 108.8 (16) |
| H121—C12—H122 | 107.5 (17) |
| C5—C12—H123 | 111.6 (16) |
| H121—C12—H123 | 108.8 (17) |
| H122—C12—H123 | 109.0 (17) |
| C9—C13—H131 | 107.8 (17) |
| C9—C13—H132 | 108.9 (16) |
| H131—C13—H132 | 110.3 (17) |
| C9—C13—H133 | 110.1 (16) |
| H131—C13—H133 | 110.1 (17) |
| H132—C13—H133 | 109.7 (17) |
| C9—C14—H141 | 109.9 (16) |
| C9—C14—H142 | 111.0 (16) |
| H141—C14—H142 | 108.6 (17) |
| C9—C14—H143 | 110.1 (16) |
| H141—C14—H143 | 107.9 (17) |
| H142—C14—H143 | 109.2 (17) |
| B7—C20—B8 | 62.2 (4) |
| B7—C20—B9 | 114.7 (4) |
| B8—C20—B9 | 63.3 (3) |
| B7—C20—B10 | 115.2 (4) |
| B8—C20—B10 | 115.2 (4) |
| B9—C20—B10 | 62.6 (3) |
| B7—C20—B11 | 63.3 (4) |
| B8—C20—B11 | 115.1 (4) |
| B9—C20—B11 | 114.8 (4) |
| B10—C20—B11 | 62.8 (3) |
| B7—C20—H20 | 117.5 |
| B8—C20—H20 | 117.5 |
| B9—C20—H20 | 117.8 |
| B10—C20—H20 | 117.5 |
| B11—C20—H20 | 117.3 |
| Cl1—C51—C52 | 111.7 (4) |
| Cl1—C51—H511 | 108.9 |
| C52—C51—H511 | 109.3 |
| Cl1—C51—H512 | 108.9 |
| C52—C51—H512 | 108.6 |
| H511—C51—H512 | 109.5 |
| C51—C52—Cl2 | 113.4 (4) |
| C51—C52—H521 | 108.0 |
| Cl2—C52—H521 | 108.2 |
| C51—C52—H522 | 108.9 |
| Cl2—C52—H522 | 108.8 |
| H521—C52—H522 | 109.5 |
| Br1—B1—B2 | 121.9 (4) |
| Br1—B1—B3 | 121.7 (4) |
| B2—B1—B3 | 59.3 (3) |
| Br1—B1—B4 | 121.8 (4) |
| B2—B1—B4 | 107.5 (4) |
| B3—B1—B4 | 60.0 (3) |
| Br1—B1—B5 | 122.2 (4) |
| B2—B1—B5 | 107.8 (4) |
| B3—B1—B5 | 107.6 (4) |
| B4—B1—B5 | 59.8 (3) |
| Br1—B1—B6 | 122.1 (4) |
| B2—B1—B6 | 60.1 (3) |
| B3—B1—B6 | 107.5 (4) |
| B4—B1—B6 | 107.8 (4) |
| B5—B1—B6 | 60.1 (3) |
| B1—B2—Br2 | 121.2 (4) |
| B1—B2—B3 | 60.0 (3) |
| Br2—B2—B3 | 121.8 (4) |
| B1—B2—B6 | 59.8 (3) |
| Br2—B2—B6 | 121.4 (4) |
| B3—B2—B6 | 107.9 (4) |
| B1—B2—B7 | 107.2 (4) |
| Br2—B2—B7 | 122.9 (4) |
| B3—B2—B7 | 107.2 (5) |
| B6—B2—B7 | 59.7 (3) |
| B1—B2—B8 | 107.9 (4) |
| Br2—B2—B8 | 122.5 (4) |
| B3—B2—B8 | 60.0 (3) |
| B6—B2—B8 | 107.7 (4) |
| B7—B2—B8 | 59.5 (4) |
| B1—B3—B2 | 60.7 (3) |
| B1—B3—Br3 | 120.9 (4) |
| B2—B3—Br3 | 121.8 (4) |
| B1—B3—B4 | 60.1 (3) |
| B2—B3—B4 | 108.6 (4) |
| Br3—B3—B4 | 121.0 (4) |
| B1—B3—B8 | 107.8 (4) |
| B2—B3—B8 | 59.4 (3) |
| Br3—B3—B8 | 122.8 (4) |
| B4—B3—B8 | 107.9 (4) |
| B1—B3—B9 | 108.0 (4) |
| B2—B3—B9 | 108.3 (4) |
| Br3—B3—B9 | 121.8 (4) |
| B4—B3—B9 | 59.6 (3) |
| B8—B3—B9 | 60.5 (3) |
| B1—B4—B3 | 59.9 (3) |
| B1—B4—Br4 | 122.9 (4) |
| B3—B4—Br4 | 121.9 (4) |
| B1—B4—B5 | 60.3 (3) |
| B3—B4—B5 | 108.0 (4) |
| Br4—B4—B5 | 122.5 (4) |
| B1—B4—B9 | 108.0 (4) |
| B3—B4—B9 | 59.8 (3) |
| Br4—B4—B9 | 120.4 (4) |
| B5—B4—B9 | 107.9 (4) |
| B1—B4—B10 | 108.2 (4) |
| B3—B4—B10 | 107.7 (4) |
| Br4—B4—B10 | 120.9 (4) |
| B5—B4—B10 | 59.9 (3) |
| B9—B4—B10 | 60.0 (3) |
| B1—B5—B4 | 59.9 (3) |
| B1—B5—Br5 | 122.3 (4) |
| B4—B5—Br5 | 122.0 (4) |
| B1—B5—B6 | 59.9 (3) |
| B4—B5—B6 | 107.8 (4) |
| Br5—B5—B6 | 121.9 (4) |
| B1—B5—B10 | 107.9 (4) |
| B4—B5—B10 | 59.9 (3) |
| Br5—B5—B10 | 121.4 (4) |
| B6—B5—B10 | 108.0 (4) |
| B1—B5—B11 | 107.5 (4) |
| B4—B5—B11 | 107.6 (4) |
| Br5—B5—B11 | 121.8 (4) |
| B6—B5—B11 | 59.9 (3) |
| B10—B5—B11 | 59.9 (3) |
| B2—B6—B5 | 107.8 (4) |
| B2—B6—B1 | 60.1 (3) |
| B5—B6—B1 | 60.1 (3) |
| B2—B6—Br6 | 123.2 (4) |
| B5—B6—Br6 | 121.5 (4) |
| B1—B6—Br6 | 122.9 (4) |
| B2—B6—B7 | 58.9 (3) |
| B5—B6—B7 | 106.9 (4) |
| B1—B6—B7 | 106.7 (4) |
| Br6—B6—B7 | 122.5 (4) |
| B2—B6—B11 | 107.1 (4) |
| B5—B6—B11 | 59.4 (3) |
| B1—B6—B11 | 107.3 (4) |
| Br6—B6—B11 | 120.9 (4) |
| B7—B6—B11 | 59.8 (3) |
| C20—B7—B6 | 105.8 (4) |
| C20—B7—B2 | 105.9 (4) |

| | | | |
|---|---|---|---|
| B6—B7—B2 | 61.4 (3) | C20—B9—B10 | 58.8 (3) |
| C20—B7—Br7 | 125.3 (11) | B8—B9—B10 | 107.7 (4) |
| B6—B7—Br7 | 118.9 (10) | B3—B9—B10 | 108.6 (4) |
| B2—B7—Br7 | 122.0 (11) | B4—B9—B10 | 60.3 (3) |
| C20—B7—B8 | 59.3 (3) | C20—B9—H9 | 123.5 |
| B6—B7—B8 | 109.6 (4) | B8—B9—H9 | 121.2 |
| B2—B7—B8 | 60.2 (4) | B3—B9—H9 | 123.7 |
| Br7—B7—B8 | 123.6 (10) | B4—B9—H9 | 123.8 |
| C20—B7—B11 | 58.8 (3) | B10—B9—H9 | 120.7 |
| B6—B7—B11 | 60.5 (3) | C20—B10—B4 | 104.0 (4) |
| B2—B7—B11 | 109.6 (4) | C20—B10—B5 | 104.0 (4) |
| Br7—B7—B11 | 118.8 (10) | B4—B10—B5 | 60.2 (3) |
| B8—B7—B11 | 108.9 (5) | C20—B10—B9 | 58.7 (3) |
| C20—B7—H7 | 122.9 | B4—B10—B9 | 59.7 (3) |
| B6—B7—H7 | 122.4 | B5—B10—B9 | 108.0 (4) |
| B2—B7—H7 | 122.4 | C20—B10—B11 | 58.1 (3) |
| B8—B7—H7 | 120.4 | B4—B10—B11 | 107.7 (4) |
| B11—B7—H7 | 120.5 | B5—B10—B11 | 59.9 (3) |
| C20—B8—B3 | 104.9 (4) | B9—B10—B11 | 107.7 (5) |
| C20—B8—B2 | 105.4 (4) | C20—B10—H10 | 123.7 |
| B3—B8—B2 | 60.6 (4) | B4—B10—H10 | 124.3 |
| C20—B8—B7 | 58.5 (3) | B5—B10—H10 | 123.4 |
| B3—B8—B7 | 108.3 (4) | B9—B10—H10 | 121.5 |
| B2—B8—B7 | 60.3 (4) | B11—B10—H10 | 120.7 |
| C20—B8—B9 | 58.8 (3) | C20—B11—B6 | 104.6 (4) |
| B3—B8—B9 | 60.0 (3) | C20—B11—B10 | 59.1 (3) |
| B2—B8—B9 | 108.9 (4) | B6—B11—B10 | 108.9 (4) |
| B7—B8—B9 | 108.0 (5) | C20—B11—B5 | 105.0 (4) |
| C20—B8—H8 | 123.1 | B6—B11—B5 | 60.7 (3) |
| B3—B8—H8 | 123.2 | B10—B11—B5 | 60.2 (3) |
| B2—B8—H8 | 123.0 | C20—B11—B7 | 57.9 (3) |
| B7—B8—H8 | 121.3 | B6—B11—B7 | 59.8 (3) |
| B9—B8—H8 | 120.5 | B10—B11—B7 | 107.7 (4) |
| C20—B9—B8 | 57.9 (3) | B5—B11—B7 | 107.9 (4) |
| C20—B9—B3 | 103.8 (4) | C20—B11—H11 | 123.4 |
| B8—B9—B3 | 59.5 (4) | B6—B11—H11 | 122.9 |
| C20—B9—B4 | 104.6 (4) | B10—B11—H11 | 120.9 |
| B8—B9—B4 | 107.8 (4) | B5—B11—H11 | 123.4 |
| B3—B9—B4 | 60.6 (3) | B7—B11—H11 | 121.3 |

| | | | |
|---|---|---|---|
| Br1—B1—B2—Br2 | −0.6 (6) | Br2—B2—B3—B1 | 110.3 (4) |
| Br1—B1—B2—B3 | 110.5 (5) | Br2—B2—B3—B4 | 147.9 (4) |
| Br1—B1—B2—B6 | −111.3 (4) | Br2—B2—B3—B8 | −111.9 (5) |
| Br1—B1—B2—B7 | −149.2 (4) | Br2—B2—B3—B9 | −149.0 (4) |
| Br1—B1—B2—B8 | 148.2 (4) | Br2—B2—B6—Br6 | 1.6 (6) |
| Br1—B1—B3—Br3 | 0.8 (6) | Br2—B2—B6—B1 | −110.4 (4) |
| Br1—B1—B3—B2 | −110.7 (4) | Br2—B2—B6—B5 | −148.2 (4) |
| Br1—B1—B3—B4 | 111.1 (5) | Br2—B2—B6—B7 | 112.4 (5) |
| Br1—B1—B3—B8 | −148.1 (4) | Br2—B2—B6—B11 | 149.1 (4) |
| Br1—B1—B3—B9 | 147.9 (4) | Br2—B2—B7—Br7 | −2 (1) |
| Br1—B1—B4—Br4 | −0.2 (6) | Br2—B2—B7—C20 | 150.5 (4) |
| Br1—B1—B4—B3 | −110.9 (5) | Br2—B2—B7—B6 | −110.0 (5) |
| Br1—B1—B4—B5 | 111.4 (4) | Br2—B2—B7—B8 | 111.2 (5) |
| Br1—B1—B4—B9 | −147.7 (4) | Br2—B2—B7—B11 | −147.6 (4) |
| Br1—B1—B4—B10 | 148.8 (4) | Br2—B2—B8—C20 | −150.6 (4) |
| Br1—B1—B5—Br5 | 0.3 (6) | Br2—B2—B8—B3 | 110.7 (5) |
| Br1—B1—B5—B4 | −110.7 (5) | Br2—B2—B8—B7 | −111.9 (5) |
| Br1—B1—B5—B6 | 111.2 (4) | Br2—B2—B8—B9 | 147.7 (4) |
| Br1—B1—B5—B10 | −148.0 (4) | Br3—B3—B1—B2 | 111.6 (4) |
| Br1—B1—B5—B11 | 148.7 (4) | Br3—B3—B1—B4 | −110.2 (4) |
| Br1—B1—B6—Br6 | −1.3 (6) | Br3—B3—B1—B5 | −147.8 (4) |
| Br1—B1—B6—B2 | 110.9 (4) | Br3—B3—B1—B6 | 148.9 (4) |
| Br1—B1—B6—B5 | −111.5 (4) | Br3—B3—B2—B1 | −110.3 (4) |
| Br1—B1—B6—B7 | 148.3 (4) | Br3—B3—B2—B6 | −147.5 (4) |
| Br1—B1—B6—B11 | −148.9 (4) | Br3—B3—B2—B7 | 149.5 (4) |
| Br2—B2—B1—B3 | −111.2 (5) | Br3—B3—B2—B8 | 111.9 (4) |
| Br2—B2—B1—B4 | −148.4 (4) | Br3—B3—B4—Br4 | −1.9 (6) |
| Br2—B2—B1—B5 | 148.5 (4) | Br3—B3—B4—B1 | 110.3 (4) |
| Br2—B2—B1—B6 | 110.7 (4) | Br3—B3—B4—B5 | 148.2 (4) |
| Br2—B2—B3—Br3 | 0.0 (6) | Br3—B3—B4—B9 | −111.1 (4) |

| | |
|---|---|
| Br3—B3—B4—B10 | −148.6 (4) |
| Br3—B3—B8—C20 | 150.2 (4) |
| Br3—B3—B8—B2 | −110.3 (4) |
| Br3—B3—B8—B7 | −148.5 (4) |
| Br3—B3—B8—B9 | 110.8 (4) |
| Br3—B3—B9—C20 | −151.2 (4) |
| Br3—B3—B9—B4 | 109.7 (5) |
| Br3—B3—B9—B8 | −112.4 (5) |
| Br3—B3—B9—B10 | 147.6 (4) |
| Br4—B4—B1—B2 | 147.6 (4) |
| Br4—B4—B1—B3 | 110.6 (5) |
| Br4—B4—B1—B5 | −111.7 (5) |
| Br4—B4—B1—B6 | −149.1 (4) |
| Br4—B4—B3—B1 | −112.2 (4) |
| Br4—B4—B3—B2 | −150.1 (4) |
| Br4—B4—B3—B8 | 147.1 (4) |
| Br4—B4—B3—B9 | 109.2 (4) |
| Br4—B4—B5—Br5 | 0.7 (6) |
| Br4—B4—B5—B1 | 112.2 (4) |
| Br4—B4—B5—B6 | 149.5 (4) |
| Br4—B4—B5—B10 | −109.7 (4) |
| Br4—B4—B5—B11 | −147.4 (4) |
| Br4—B4—B9—C20 | 150.6 (4) |
| Br4—B4—B9—B3 | −111.5 (4) |
| Br4—B4—B9—B8 | −148.9 (4) |
| Br4—B4—B9—B10 | 110.5 (4) |
| Br4—B4—B10—C20 | −149.6 (4) |
| Br4—B4—B10—B5 | 112.1 (4) |
| Br4—B4—B10—B9 | −109.6 (4) |
| Br4—B4—B10—B11 | 149.9 (4) |
| Br5—B5—B1—B2 | −148.7 (4) |
| Br5—B5—B1—B3 | 148.7 (4) |
| Br5—B5—B1—B4 | 111.0 (5) |
| Br5—B5—B1—B6 | −110.9 (4) |
| Br5—B5—B4—B1 | −111.5 (4) |
| Br5—B5—B4—B3 | −149.1 (4) |
| Br5—B5—B4—B9 | 147.7 (4) |
| Br5—B5—B4—B10 | 110.4 (4) |
| Br5—B5—B6—Br6 | −1.1 (6) |
| Br5—B5—B6—B1 | 111.5 (4) |
| Br5—B5—B6—B2 | 149.4 (4) |
| Br5—B5—B6—B7 | −148.6 (4) |
| Br5—B5—B6—B11 | −110.8 (5) |
| Br5—B5—B10—C20 | 150.3 (4) |
| Br5—B5—B10—B4 | −111.3 (5) |
| Br5—B5—B10—B9 | −148.5 (4) |
| Br5—B5—B10—B11 | 111.0 (5) |
| Br5—B5—B11—C20 | −150.4 (4) |
| Br5—B5—B11—B6 | 111.0 (4) |
| Br5—B5—B11—B7 | 149.1 (4) |
| Br5—B5—B11—B10 | −110.5 (4) |
| Br6—B6—B1—B2 | −112.2 (4) |
| Br6—B6—B1—B3 | −149.2 (4) |
| Br6—B6—B1—B4 | 147.4 (4) |
| Br6—B6—B1—B5 | 110.2 (4) |
| Br6—B6—B2—B1 | 112.0 (5) |
| Br6—B6—B2—B3 | 149.3 (4) |
| Br6—B6—B2—B7 | −110.7 (5) |
| Br6—B6—B2—B8 | −147.3 (4) |
| Br6—B6—B5—B1 | −112.5 (4) |
| Br6—B6—B5—B4 | −149.9 (4) |
| Br6—B6—B5—B10 | 146.8 (4) |
| Br6—B6—B5—B11 | 109.7 (5) |
| Br6—B6—B7—Br7 | −1 (1) |
| Br6—B6—B7—C20 | −148.3 (4) |
| Br6—B6—B7—B2 | 111.9 (4) |
| Br6—B6—B7—B8 | 149.2 (4) |
| Br6—B6—B7—B11 | −109.5 (5) |
| Br6—B6—B11—C20 | 150.1 (4) |
| Br6—B6—B11—B5 | −110.7 (4) |
| Br6—B6—B11—B7 | 112.0 (5) |
| Br6—B6—B11—B10 | −148.1 (4) |
| Br7—B7—C20—B8 | 112 (1) |
| Br7—B7—C20—B9 | 148 (1) |
| Br7—B7—C20—B10 | −142 (1) |
| Br7—B7—C20—B11 | −105 (1) |
| Br7—B7—B2—B1 | 146 (1) |
| Br7—B7—B2—B3 | −151 (1) |
| Br7—B7—B2—B6 | 108 (1) |
| Br7—B7—B2—B8 | −113 (1) |
| Br7—B7—B6—B1 | −151 (1) |
| Br7—B7—B6—B2 | −113 (1) |
| Br7—B7—B6—B5 | 146 (1) |
| Br7—B7—B6—B11 | 109 (1) |
| Br7—B7—B8—C20 | −114 (1) |
| Br7—B7—B8—B2 | 111 (1) |
| Br7—B7—B8—B3 | 149 (1) |
| Br7—B7—B8—B9 | −147 (1) |
| Br7—B7—B11—C20 | 116 (1) |
| Br7—B7—B11—B5 | −147 (1) |
| Br7—B7—B11—B6 | −109 (1) |
| Br7—B7—B11—B10 | 149 (1) |
| Cl1—C51—C52—Cl2 | 60.0 (5) |
| O1—C4—C3—C2 | 173.1 (4) |
| O1—C4—C3—C11 | −6.8 (6) |
| O1—C4—C5—C6 | −173.1 (4) |
| O1—C4—C5—C12 | 5.2 (6) |
| O2—C1—C2—C3 | −174.7 (4) |
| O2—C1—C2—C10 | 4.2 (6) |
| O2—C1—C6—C5 | 174.7 (4) |
| O2—C1—C6—C7 | −5.0 (6) |
| O2—C9—C8—C7 | −56.2 (6) |
| C1—O2—C9—C8 | 31.7 (6) |
| C1—O2—C9—C13 | 151.4 (4) |
| C1—O2—C9—C14 | −91.0 (5) |
| C1—C2—C3—C4 | 0.2 (6) |
| C1—C2—C3—C11 | −179.9 (4) |
| C1—C6—C5—C4 | −0.1 (6) |
| C1—C6—C5—C12 | −178.3 (4) |
| C1—C6—C7—C8 | −20.4 (6) |
| C2—C1—O2—C9 | 179.4 (4) |
| C2—C1—C6—C5 | −6.0 (7) |
| C2—C1—C6—C7 | 174.4 (4) |
| C2—C3—C4—C5 | −5.8 (6) |
| C3—C2—C1—C6 | 5.9 (7) |
| C3—C4—C5—C6 | 5.7 (6) |
| C3—C4—C5—C12 | −175.9 (4) |
| C4—C3—C2—C10 | −178.6 (4) |
| C4—C5—C6—C7 | 179.6 (4) |
| C5—C4—C3—C11 | 174.3 (4) |
| C5—C6—C7—C8 | 159.9 (4) |
| C6—C1—O2—C9 | −1.1 (7) |
| C6—C1—C2—C10 | −175.2 (4) |
| C6—C7—C8—C9 | 51.9 (6) |
| C7—C6—C5—C12 | 1.4 (7) |
| C7—C8—C9—C13 | −167.9 (5) |
| C7—C8—C9—C14 | 59.5 (6) |
| C10—C2—C3—C11 | 1.4 (8) |
| C20—B7—B2—B1 | −61.7 (5) |
| C20—B7—B2—B3 | 1.4 (5) |
| C20—B7—B2—B6 | −99.5 (4) |
| C20—B7—B2—B8 | 39.2 (4) |
| C20—B7—B6—B1 | 61.9 (5) |
| C20—B7—B6—B2 | 99.9 (4) |
| C20—B7—B6—B5 | −1.1 (5) |
| C20—B7—B6—B11 | −38.8 (4) |
| C20—B7—B8—B2 | −135.0 (4) |
| C20—B7—B8—B3 | −96.6 (4) |

| | |
|---|---|
| C20—B7—B8—B9 | −33.2 (4) |
| C20—B7—B11—B5 | 96.8 (4) |
| C20—B7—B11—B6 | 135.2 (4) |
| C20—B7—B11—B10 | 33.2 (4) |
| C20—B8—B2—B1 | 61.1 (5) |
| C20—B8—B2—B3 | 98.7 (4) |
| C20—B8—B2—B6 | −2.0 (5) |
| C20—B8—B2—B7 | −38.7 (4) |
| C20—B8—B3—B1 | −61.5 (5) |
| C20—B8—B3—B2 | −99.5 (4) |
| C20—B8—B3—B4 | 2.0 (5) |
| C20—B8—B3—B9 | 39.4 (4) |
| C20—B8—B7—B2 | 135.0 (4) |
| C20—B8—B7—B6 | 97.2 (4) |
| C20—B8—B7—B11 | 32.7 (4) |
| C20—B8—B9—B3 | −134.2 (4) |
| C20—B8—B9—B4 | −96.2 (4) |
| C20—B8—B9—B10 | −32.6 (4) |
| C20—B9—B3—B1 | 62.1 (5) |
| C20—B9—B3—B2 | −2.2 (5) |
| C20—B9—B3—B4 | 99.1 (4) |
| C20—B9—B3—B8 | −38.7 (4) |
| C20—B9—B4—B1 | −61.0 (5) |
| C20—B9—B4—B3 | −97.9 (4) |
| C20—B9—B4—B5 | 2.8 (5) |
| C20—B9—B4—B10 | 40.1 (4) |
| C20—B9—B8—B2 | 97.0 (5) |
| C20—B9—B8—B3 | 134.2 (4) |
| C20—B9—B8—B7 | 33.0 (4) |
| C20—B9—B10—B4 | −133.2 (4) |
| C20—B9—B10—B5 | −95.8 (4) |
| C20—B9—B10—B11 | −32.5 (4) |
| C20—B10—B4—B1 | 60.7 (5) |
| C20—B10—B4—B3 | −2.5 (5) |
| C20—B10—B4—B5 | 98.3 (4) |
| C20—B10—B4—B9 | −39.9 (4) |
| C20—B10—B5—B1 | −61.1 (5) |
| C20—B10—B5—B4 | −98.4 (4) |
| C20—B10—B5—B6 | 2.2 (5) |
| C20—B10—B5—B11 | 39.3 (4) |
| C20—B10—B9—B3 | 95.2 (4) |
| C20—B10—B9—B4 | 133.2 (4) |
| C20—B10—B9—B8 | 32.3 (4) |
| C20—B10—B11—B5 | −133.7 (4) |
| C20—B10—B11—B6 | −96.1 (4) |
| C20—B10—B11—B7 | −32.8 (4) |
| C20—B11—B5—B1 | 61.0 (5) |
| C20—B11—B5—B4 | −2.2 (5) |
| C20—B11—B5—B6 | 98.6 (4) |
| C20—B11—B5—B10 | −39.9 (4) |
| C20—B11—B6—B1 | −61.6 (5) |
| C20—B11—B6—B2 | 1.8 (5) |
| C20—B11—B6—B5 | −99.2 (4) |
| C20—B11—B6—B7 | 38.1 (4) |
| C20—B11—B7—B2 | −97.2 (4) |
| C20—B11—B7—B6 | −135.2 (4) |
| C20—B11—B7—B8 | −33.0 (4) |
| C20—B11—B10—B4 | 95.8 (4) |
| C20—B11—B10—B5 | 133.7 (4) |
| C20—B11—B10—B9 | 32.8 (4) |
| B1—B2—B3—B4 | 37.6 (4) |
| B1—B2—B3—B8 | 137.8 (4) |
| B1—B2—B3—B9 | 100.7 (4) |
| B1—B2—B6—B5 | −37.9 (4) |
| B1—B2—B6—B7 | −137.2 (4) |
| B1—B2—B6—B11 | −100.5 (4) |
| B1—B2—B7—B6 | 37.9 (4) |
| B1—B2—B7—B8 | −100.9 (4) |
| B1—B2—B7—B11 | 0.3 (5) |
| B1—B2—B8—B3 | −37.6 (4) |
| B1—B2—B8—B7 | 99.8 (4) |
| B1—B2—B8—B9 | −0.7 (6) |
| B1—B3—B2—B6 | −37.2 (4) |
| B1—B3—B2—B7 | −100.2 (4) |
| B1—B3—B2—B8 | −137.8 (4) |
| B1—B3—B4—B5 | 37.9 (4) |
| B1—B3—B4—B9 | 138.6 (4) |
| B1—B3—B4—B10 | 101.1 (4) |
| B1—B3—B8—B2 | 38.0 (4) |
| B1—B3—B8—B7 | −0.3 (5) |
| B1—B3—B8—B9 | −100.9 (4) |
| B1—B3—B9—B4 | −37.0 (4) |
| B1—B3—B9—B8 | 100.8 (4) |
| B1—B3—B9—B10 | 0.8 (5) |
| B1—B4—B3—B2 | −37.8 (4) |
| B1—B4—B3—B8 | −100.7 (4) |
| B1—B4—B3—B9 | −138.6 (4) |
| B1—B4—B5—B6 | 37.3 (4) |
| B1—B4—B5—B10 | 138.2 (4) |
| B1—B4—B5—B11 | 100.5 (4) |
| B1—B4—B9—B3 | 36.9 (4) |
| B1—B4—B9—B8 | −0.5 (5) |
| B1—B4—B9—B10 | −101.0 (4) |
| B1—B4—B10—B5 | −37.5 (4) |
| B1—B4—B10—B9 | 100.7 (4) |
| B1—B4—B10—B11 | 0.2 (5) |
| B1—B5—B4—B3 | −37.7 (4) |
| B1—B5—B4—B9 | −100.8 (4) |
| B1—B5—B4—B10 | −138.2 (4) |
| B1—B5—B6—B2 | 37.9 (4) |
| B1—B5—B6—B7 | 99.9 (4) |
| B1—B5—B6—B11 | 137.7 (4) |
| B1—B5—B10—B4 | 37.3 (4) |
| B1—B5—B10—B9 | 0.1 (5) |
| B1—B5—B10—B11 | −100.3 (4) |
| B1—B5—B11—B6 | −37.6 (4) |
| B1—B5—B11—B7 | 0.4 (5) |
| B1—B5—B11—B10 | 100.9 (4) |
| B1—B6—B2—B3 | 37.3 (4) |
| B1—B6—B2—B7 | 137.2 (4) |
| B1—B6—B2—B8 | 100.7 (4) |
| B1—B6—B5—B4 | −37.4 (4) |
| B1—B6—B5—B10 | −100.7 (4) |
| B1—B6—B5—B11 | −137.7 (4) |
| B1—B6—B7—B2 | −38.0 (4) |
| B1—B6—B7—B8 | −0.6 (5) |
| B1—B6—B7—B11 | 100.7 (4) |
| B1—B6—B11—B5 | 37.6 (4) |
| B1—B6—B11—B7 | −99.6 (4) |
| B1—B6—B11—B10 | 0.2 (5) |
| B2—B1—B3—B4 | −138.2 (4) |
| B2—B1—B3—B8 | −37.4 (4) |
| B2—B1—B3—B9 | −101.4 (4) |
| B2—B1—B4—B3 | 36.9 (4) |
| B2—B1—B4—B5 | −100.8 (4) |
| B2—B1—B4—B9 | 0.1 (5) |
| B2—B1—B4—B10 | −63.4 (5) |
| B2—B1—B5—B4 | 100.3 (4) |
| B2—B1—B5—B6 | −37.9 (4) |
| B2—B1—B5—B10 | 63.0 (5) |
| B2—B1—B5—B11 | −0.3 (5) |
| B2—B1—B6—B5 | 137.6 (4) |
| B2—B1—B6—B7 | 37.4 (4) |
| B2—B1—B6—B11 | 100.2 (4) |
| B2—B3—B1—B4 | 138.2 (4) |
| B2—B3—B1—B5 | 100.6 (4) |
| B2—B3—B1—B6 | 37.3 (4) |
| B2—B3—B4—B5 | 0.0 (5) |

| | |
|---|---|
| B2—B3—B4—B9 | 100.8 (4) |
| B2—B3—B4—B10 | 63.2 (5) |
| B2—B3—B8—B7 | −38.3 (4) |
| B2—B3—B8—B9 | −138.9 (4) |
| B2—B3—B9—B4 | −101.3 (4) |
| B2—B3—B9—B8 | 36.5 (4) |
| B2—B3—B9—B10 | −63.4 (5) |
| B2—B6—B1—B3 | −37.0 (4) |
| B2—B6—B1—B4 | −100.3 (4) |
| B2—B6—B1—B5 | −137.6 (4) |
| B2—B6—B5—B4 | 0.6 (5) |
| B2—B6—B5—B10 | −62.7 (5) |
| B2—B6—B5—B11 | −99.8 (4) |
| B2—B6—B7—B8 | 37.4 (4) |
| B2—B6—B7—B11 | 138.6 (4) |
| B2—B6—B11—B5 | 101.0 (4) |
| B2—B6—B11—B7 | −36.3 (4) |
| B2—B6—B11—B10 | 63.6 (5) |
| B2—B7—C20—B8 | −39.7 (4) |
| B2—B7—C20—B9 | −3.0 (6) |
| B2—B7—C20—B10 | 66.8 (5) |
| B2—B7—C20—B11 | 103.6 (4) |
| B2—B7—B6—B5 | −101.0 (4) |
| B2—B7—B6—B11 | −138.6 (4) |
| B2—B7—B8—B3 | 38.4 (4) |
| B2—B7—B8—B9 | 101.9 (4) |
| B2—B7—B11—B5 | −0.4 (5) |
| B2—B7—B11—B6 | 38.0 (4) |
| B2—B7—B11—B10 | −64.0 (5) |
| B2—B8—C20—B7 | 39.6 (4) |
| B2—B8—C20—B9 | −103.0 (4) |
| B2—B8—C20—B10 | −66.9 (5) |
| B2—B8—C20—B11 | 3.4 (6) |
| B2—B8—B3—B4 | 101.5 (4) |
| B2—B8—B3—B9 | 138.9 (4) |
| B2—B8—B7—B6 | −37.9 (4) |
| B2—B8—B7—B11 | −102.3 (4) |
| B2—B8—B9—B3 | −37.2 (4) |
| B2—B8—B9—B4 | 0.7 (6) |
| B2—B8—B9—B10 | 64.3 (5) |
| B3—B1—B2—B6 | 138.2 (4) |
| B3—B1—B2—B7 | 100.3 (4) |
| B3—B1—B2—B8 | 37.7 (4) |
| B3—B1—B4—B5 | −137.7 (4) |
| B3—B1—B4—B9 | −36.9 (4) |
| B3—B1—B4—B10 | −100.4 (4) |
| B3—B1—B5—B4 | 37.7 (4) |
| B3—B1—B5—B6 | −100.4 (4) |
| B3—B1—B5—B10 | 0.4 (5) |
| B3—B1—B5—B11 | −62.9 (5) |
| B3—B1—B6—B5 | 100.6 (4) |
| B3—B1—B6—B7 | 0.4 (5) |
| B3—B1—B6—B11 | 63.3 (5) |
| B3—B2—B1—B4 | −37.3 (4) |
| B3—B2—B1—B5 | −100.3 (4) |
| B3—B2—B1—B6 | −138.2 (4) |
| B3—B2—B6—B5 | −0.5 (5) |
| B3—B2—B6—B7 | −99.9 (4) |
| B3—B2—B6—B11 | −63.2 (5) |
| B3—B2—B7—B6 | 100.9 (4) |
| B3—B2—B7—B8 | −37.8 (4) |
| B3—B2—B7—B11 | 63.3 (5) |
| B3—B2—B8—B7 | 137.4 (4) |
| B3—B2—B8—B9 | 36.9 (4) |
| B3—B4—B1—B5 | 137.7 (4) |
| B3—B4—B1—B6 | 100.3 (4) |
| B3—B4—B5—B6 | −0.4 (5) |
| B3—B4—B5—B10 | 100.5 (4) |
| B3—B4—B5—B11 | 62.8 (5) |
| B3—B4—B9—B8 | −37.4 (4) |
| B3—B4—B9—B10 | −137.9 (4) |
| B3—B4—B10—B5 | −100.8 (4) |
| B3—B4—B10—B9 | 37.4 (4) |
| B3—B4—B10—B11 | −63.1 (5) |
| B3—B8—C20—B7 | 102.6 (4) |
| B3—B8—C20—B9 | −40.0 (4) |
| B3—B8—C20—B10 | −3.9 (5) |
| B3—B8—C20—B11 | 66.5 (5) |
| B3—B8—B2—B6 | −100.7 (4) |
| B3—B8—B2—B7 | −137.4 (4) |
| B3—B8—B7—B6 | 0.5 (5) |
| B3—B8—B7—B11 | −63.9 (5) |
| B3—B8—B9—B4 | 37.9 (4) |
| B3—B8—B9—B10 | 101.6 (4) |
| B3—B9—C20—B7 | 3.3 (6) |
| B3—B9—C20—B8 | 39.5 (4) |
| B3—B9—C20—B10 | −103.6 (4) |
| B3—B9—C20—B11 | −67.3 (5) |
| B3—B9—B4—B5 | 100.7 (4) |
| B3—B9—B4—B10 | 137.9 (4) |
| B3—B9—B8—B7 | −101.2 (4) |
| B3—B9—B10—B4 | −38.0 (4) |
| B3—B9—B10—B5 | −0.6 (5) |
| B3—B9—B10—B11 | 62.7 (5) |
| B4—B1—B2—B6 | 100.9 (4) |
| B4—B1—B2—B7 | 63.0 (5) |
| B4—B1—B2—B8 | 0.4 (5) |
| B4—B1—B3—B8 | 100.8 (4) |
| B4—B1—B3—B9 | 36.8 (4) |
| B4—B1—B5—B6 | −138.2 (4) |
| B4—B1—B5—B10 | −37.3 (4) |
| B4—B1—B5—B11 | −100.6 (4) |
| B4—B1—B6—B5 | 37.3 (4) |
| B4—B1—B6—B7 | −63.0 (5) |
| B4—B1—B6—B11 | −0.1 (5) |
| B4—B3—B1—B5 | −37.6 (4) |
| B4—B3—B1—B6 | −100.9 (4) |
| B4—B3—B2—B6 | 0.3 (5) |
| B4—B3—B2—B7 | −62.7 (5) |
| B4—B3—B2—B8 | −100.2 (4) |
| B4—B3—B8—B7 | 63.2 (5) |
| B4—B3—B8—B9 | −37.5 (4) |
| B4—B3—B9—B8 | 137.8 (4) |
| B4—B3—B9—B10 | 37.9 (4) |
| B4—B5—B1—B6 | 138.2 (4) |
| B4—B5—B6—B7 | 62.6 (5) |
| B4—B5—B6—B11 | 100.4 (4) |
| B4—B5—B10—B9 | −37.2 (4) |
| B4—B5—B10—B11 | −137.7 (4) |
| B4—B5—B11—B6 | −100.8 (4) |
| B4—B5—B11—B7 | −62.8 (5) |
| B4—B5—B11—B10 | 37.7 (4) |
| B4—B9—C20—B7 | 66.0 (5) |
| B4—B9—C20—B8 | 102.3 (4) |
| B4—B9—C20—B10 | −40.8 (4) |
| B4—B9—C20—B11 | −4.5 (5) |
| B4—B9—B3—B8 | −137.8 (4) |
| B4—B9—B8—B7 | −63.2 (5) |
| B4—B9—B10—B5 | 37.4 (4) |
| B4—B9—B10—B11 | 100.7 (4) |
| B4—B10—C20—B7 | −65.5 (5) |
| B4—B10—C20—B8 | 4.1 (5) |
| B4—B10—C20—B9 | 40.4 (4) |
| B4—B10—C20—B11 | −102.4 (4) |
| B4—B10—B5—B6 | 100.6 (4) |
| B4—B10—B5—B11 | 137.7 (4) |
| B4—B10—B9—B8 | −100.9 (4) |
| B4—B10—B11—B5 | −37.9 (4) |

| | |
|---|---|
| B4—B10—B11—B6 | −0.3 (5) |
| B4—B10—B11—B7 | 63.0 (5) |
| B5—B1—B2—B6 | 37.9 (4) |
| B5—B1—B2—B7 | 0.0 (5) |
| B5—B1—B2—B8 | −62.6 (5) |
| B5—B1—B3—B8 | 63.2 (5) |
| B5—B1—B3—B9 | −0.8 (5) |
| B5—B1—B4—B9 | 100.8 (4) |
| B5—B1—B4—B10 | 37.4 (4) |
| B5—B1—B6—B7 | −100.2 (4) |
| B5—B1—B6—B11 | −37.4 (4) |
| B5—B4—B1—B6 | −37.4 (4) |
| B5—B4—B3—B8 | −62.8 (5) |
| B5—B4—B3—B9 | −100.7 (4) |
| B5—B4—B9—B8 | 63.2 (5) |
| B5—B4—B9—B10 | −37.3 (4) |
| B5—B4—B10—B9 | 138.2 (4) |
| B5—B4—B10—B11 | 37.7 (4) |
| B5—B6—B2—B7 | 99.4 (4) |
| B5—B6—B2—B8 | 62.8 (5) |
| B5—B6—B7—B8 | −63.6 (5) |
| B5—B6—B7—B11 | 37.7 (4) |
| B5—B6—B11—B7 | −137.3 (4) |
| B5—B6—B11—B10 | −37.4 (4) |
| B5—B10—C20—B7 | −3.2 (5) |
| B5—B10—C20—B8 | 66.4 (5) |
| B5—B10—C20—B9 | 102.7 (4) |
| B5—B10—C20—B11 | −40.2 (4) |
| B5—B10—B4—B9 | −138.2 (4) |
| B5—B10—B9—B8 | −63.5 (5) |
| B5—B10—B11—B6 | 37.6 (4) |
| B5—B10—B11—B7 | 100.9 (4) |
| B5—B11—C20—B7 | −102.0 (4) |
| B5—B11—C20—B8 | −66.3 (5) |
| B5—B11—C20—B9 | 4.3 (5) |
| B5—B11—C20—B10 | 40.5 (4) |
| B5—B11—B6—B7 | 137.3 (4) |
| B5—B11—B7—B6 | −38.4 (4) |
| B5—B11—B7—B8 | 63.8 (5) |
| B5—B11—B10—B9 | −100.9 (4) |
| B6—B1—B2—B7 | −37.9 (4) |
| B6—B1—B2—B8 | −100.5 (4) |
| B6—B1—B3—B8 | −0.1 (5) |
| B6—B1—B3—B9 | −64.1 (5) |
| B6—B1—B4—B9 | 63.4 (5) |
| B6—B1—B4—B10 | −0.0 (5) |
| B6—B1—B5—B10 | 100.8 (4) |
| B6—B1—B5—B11 | 37.6 (4) |
| B6—B2—B3—B8 | 100.6 (4) |
| B6—B2—B3—B9 | 63.5 (5) |
| B6—B2—B7—B8 | −138.8 (4) |
| B6—B2—B7—B11 | −37.6 (4) |
| B6—B2—B8—B7 | 36.7 (4) |
| B6—B2—B8—B9 | −63.8 (5) |
| B6—B5—B4—B9 | −63.5 (5) |
| B6—B5—B4—B10 | −100.9 (4) |
| B6—B5—B10—B9 | 63.4 (5) |
| B6—B5—B10—B11 | −37.0 (4) |
| B6—B5—B11—B7 | 38.0 (4) |
| B6—B5—B11—B10 | 138.5 (4) |
| B6—B7—C20—B8 | −103.7 (4) |
| B6—B7—C20—B9 | −67.1 (5) |
| B6—B7—C20—B10 | 2.8 (5) |
| B6—B7—C20—B11 | 39.6 (4) |
| B6—B7—B2—B8 | 138.8 (4) |
| B6—B7—B8—B9 | 64.0 (5) |
| B6—B7—B11—B10 | −102.0 (4) |
| B6—B11—C20—B7 | −39.0 (4) |
| B6—B11—C20—B8 | −3.3 (5) |
| B6—B11—C20—B9 | 67.3 (5) |
| B6—B11—C20—B10 | 103.5 (4) |
| B6—B11—B5—B10 | −138.5 (4) |
| B6—B11—B7—B8 | 102.2 (5) |
| B6—B11—B10—B9 | −63.3 (5) |
| B7—C20—B8—B9 | 142.6 (4) |
| B7—C20—B9—B8 | −36.2 (4) |
| B7—C20—B9—B10 | 106.9 (5) |
| B7—C20—B10—B9 | −105.9 (4) |
| B7—C20—B10—B11 | 37.0 (4) |
| B7—C20—B11—B10 | −142.5 (4) |
| B7—B2—B3—B8 | 37.6 (4) |
| B7—B2—B3—B9 | 0.5 (5) |
| B7—B2—B6—B11 | 36.7 (4) |
| B7—B2—B8—B9 | −100.5 (4) |
| B7—B6—B2—B8 | −36.6 (4) |
| B7—B6—B5—B10 | −0.7 (5) |
| B7—B6—B5—B11 | −37.8 (4) |
| B7—B6—B11—B10 | 99.9 (4) |
| B7—B8—C20—B9 | −142.6 (4) |
| B7—B8—C20—B10 | −106.5 (5) |
| B7—B8—C20—B11 | −36.1 (4) |
| B7—B8—B3—B9 | 100.7 (4) |
| B7—B8—B9—B10 | 0.4 (5) |
| B7—B11—C20—B8 | 35.7 (4) |
| B7—B11—C20—B9 | 106.3 (5) |
| B7—B11—C20—B10 | 142.5 (4) |
| B7—B11—B5—B10 | −100.5 (4) |
| B7—B11—B10—B9 | −0.0 (5) |
| B8—C20—B7—B11 | −143.3 (4) |
| B8—C20—B9—B10 | 143.1 (4) |
| B8—C20—B10—B9 | −36.4 (4) |
| B8—C20—B10—B11 | 106.5 (5) |
| B8—C20—B11—B10 | −106.8 (4) |
| B8—B2—B3—B9 | −37.1 (4) |
| B8—B2—B6—B11 | 0.2 (5) |
| B8—B2—B7—B11 | 101.2 (4) |
| B8—B3—B4—B9 | 37.9 (4) |
| B8—B3—B4—B10 | 0.4 (5) |
| B8—B3—B9—B10 | −100.0 (4) |
| B8—B7—C20—B9 | 36.6 (4) |
| B8—B7—C20—B10 | 106.5 (4) |
| B8—B7—C20—B11 | 143.3 (4) |
| B8—B7—B6—B11 | −101.3 (4) |
| B8—B7—B11—B10 | 0.3 (5) |
| B8—B9—C20—B10 | −143.1 (4) |
| B8—B9—C20—B11 | −106.8 (5) |
| B8—B9—B4—B10 | 100.5 (4) |
| B8—B9—B10—B11 | −0.2 (5) |
| B9—C20—B7—B11 | −106.6 (5) |
| B9—C20—B10—B11 | 142.9 (4) |
| B9—C20—B11—B10 | −36.2 (4) |
| B9—B3—B4—B10 | −37.5 (4) |
| B9—B4—B5—B10 | 37.3 (4) |
| B9—B4—B5—B11 | −0.4 (5) |
| B9—B4—B10—B11 | −100.5 (4) |
| B9—B8—C20—B10 | 36.1 (4) |
| B9—B8—C20—B11 | 106.4 (5) |
| B9—B8—B7—B11 | −0.4 (5) |
| B9—B10—C20—B11 | −142.9 (4) |
| B9—B10—B5—B11 | 100.4 (4) |
| B10—C20—B7—B11 | −36.8 (4) |
| B10—B4—B5—B11 | −37.7 (4) |
| B10—B5—B6—B11 | 37.1 (4) |
| B10—B9—C20—B11 | 36.3 (4) |

Table *S*4. *Contact distances (Å)*

| | | | |
|---|---|---|---|
| Br4···C5 | 3.489 (4) | Cl2···Cl2$^{v}$ | 3.457 (3) |
| Br4···C6 | 3.550 (4) | Cl2···C14 | 3.596 (6) |
| Br5···C4$^{i}$ | 3.403 (4) | O1···B11$^{vi}$ | 3.417 (7) |
| Br7···C11$^{ii}$ | 3.29 (2) | O1···C20$^{vi}$ | 3.437 (6) |
| Br7···C51$^{iii}$ | 3.31 (3) | O1···C12$^{vii}$ | 3.489 (7) |
| Br7···C12$^{iv}$ | 3.46 (3) | O1···C7$^{vii}$ | 3.526 (6) |

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

**Figure Captions for $(C_{14}H_{19}O_2)^+(CH_6B_{11}Br_6)^- \bullet C_2H_4Cl_2$**

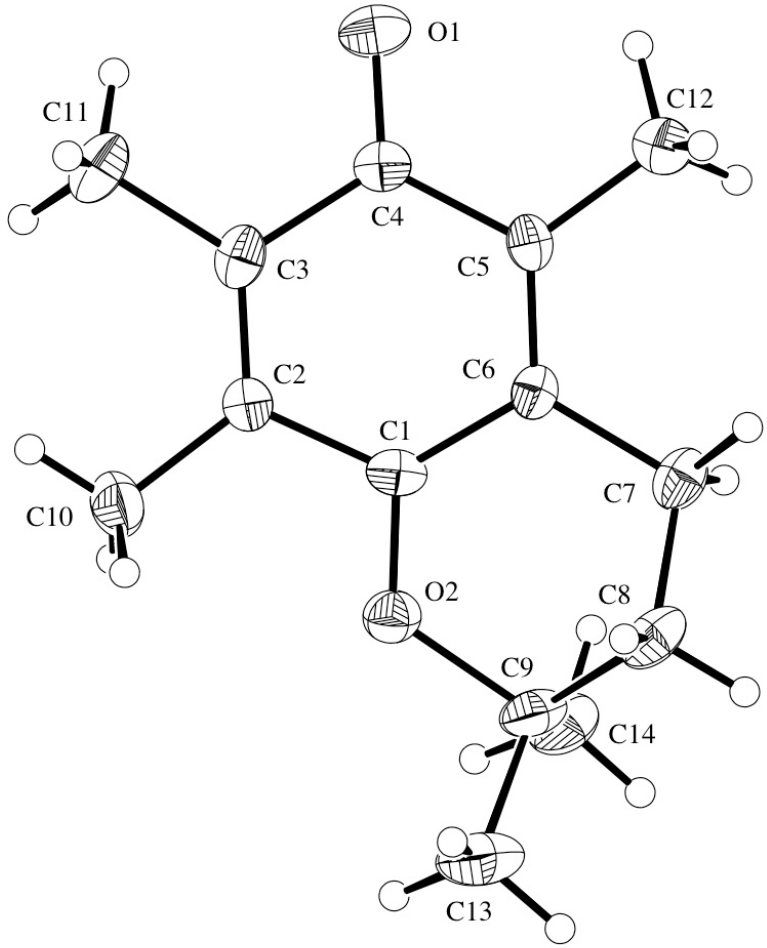


**Figure 1**. Molecular structure of the cation of $(C_{14}H_{19}O_2)^+(CH_6B_{11}Br_6)^- \bullet C_2H_4Cl_2$ with labelling of selected atoms. Anisotropic displacement ellipsoids show 30% probability levels. Hydrogen atoms are drawn as circles with small radii.

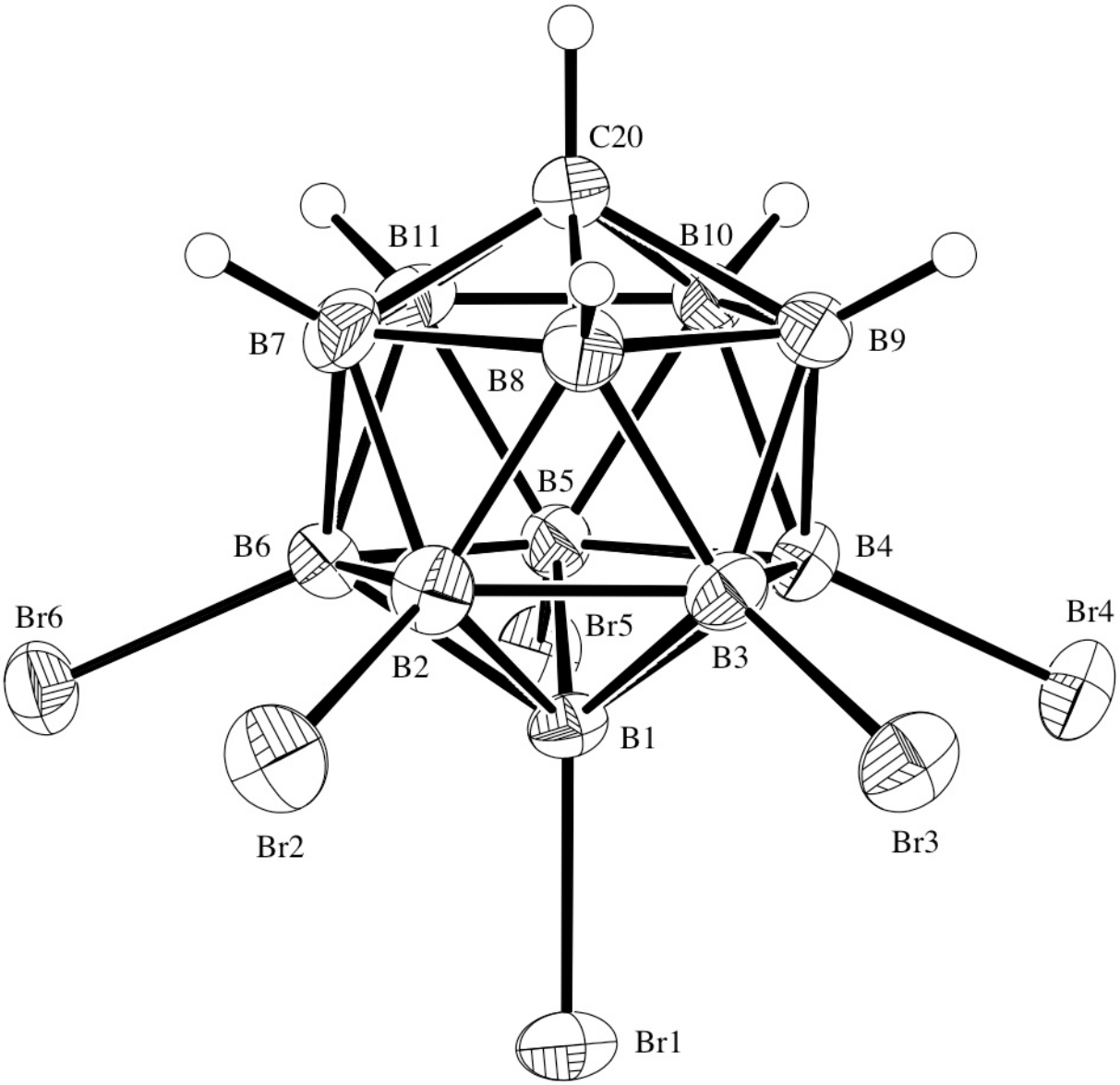


**Figure 2**. Molecular structure of the anion of $(C_{14}H_{19}O_2)^+(CH_6B_{11}Br_6)^- \bullet C_2H_4Cl_2$ with labelling of selected atoms. Anisotropic displacement ellipsoids show 30% probability levels. Hydrogen atoms are drawn as circles with small radii. The low-ocupancy site Br7 is not shown.

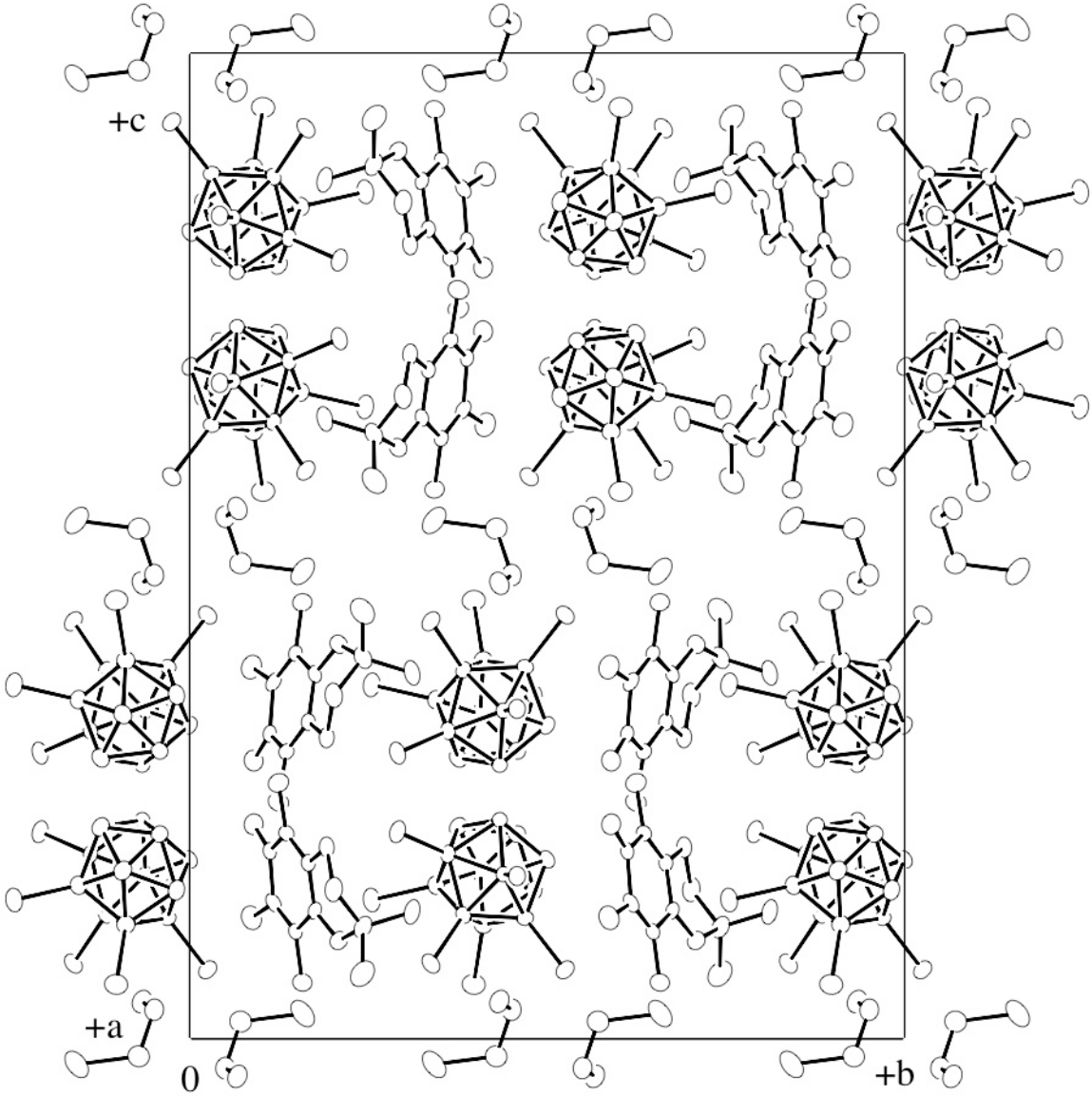


**Figure 3**. Unit cell packing diagram of $(C_{14}H_{19}O_2)^+(CH_6B_{11}Br_6)^- \bullet C_2H_4Cl_2$ projected down the *a* axis. Hydrogen atoms and the low-ocupancy site Br7 are not shown.

31 Mar 2006

## Crystal structure of $(C_{14}H_{19}O_2)^+(B(C_6F_5)_4)^-$ –web0602

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### Abstract

The crystal structure of $(C_{14}H_{19}O_2)^+(B(C_6F_5)_4)^-$ is reported.

### Comment

The space group is noncentrosymmetric but the anomolous dispersion terms are very small for all elements in the structure, so the absolute structure of the crystal can not be determined in this experiment. Consequently Friedel-pair reflections have been averaged and the Flack parameter has not been refined. The space group is not enantiomorphic so the compound is present as a racemate within the crystal structure.

The crystallographic asymmetric unit consists of one $(C_{14}H_{19}O_2)^+$ cation and one $(B(C_6F_5)_4)^-$ anion.

The largest peaks in the final difference map are all of near-equal intensity and have no chemical significance.

### Experimental

The compound was prepared by SBL and recrystallized from dichloroethane. The sample ID is RDW02.

*Crystal data*

$C_{14}H_{19}O_2^+.C_{24}BF_{20}^-$
$M_r = 898.34$
Monoclinic
$Cc$
$a = 11.9549\,(2)$ Å
$b = 17.4980\,(4)$ Å
$c = 17.3301\,(3)$ Å
$\beta = 98.7693\,(13)°$
$V = 3582.85\,(12)$ Å$^3$
$Z = 4$
$D_x = 1.665$ Mg m$^{-3}$
$D_m$ not measured
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 29935 reflections
$\theta = 3$–$27°$
$\mu = 0.173$ mm$^{-1}$
$T = 200$ K
Plate
Red
$0.31 \times 0.20 \times 0.07$ mm
Crystal source: local

*Data collection*

Nonius KappaCCD diffractometer
$\varphi$ and $\omega$ scans with CCD
Absorption correction:
multi-scan Denzo/Scalepack (Otwinowski & Minor, 1997)
$T_{\min} = 0.87$, $T_{\max} = 0.99$
38073 measured reflections
4113 independent reflections

3377 reflections with
$I > 2.0\sigma(I)$
$R_{\rm int} = 0.032$
$\theta_{\max} = 27.5°$
$h = -15 \rightarrow 15$
$k = -22 \rightarrow 22$
$l = -22 \rightarrow 22$

*Refinement*

Refinement on $F^2$
$R(F) = 0.0316$
$wR(F^2) = 0.0733$
$S = 0.8816$
4104 reflections
607 parameters
Only coordinates of H atoms refined

Method= Modified Sheldrick $w$=1/[$\sigma^2(F^2)$ + ( 0.05$P$)$^2$ + 1.28$P$] ,
where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.038298$
$\Delta\rho_{\max} = 0.24$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.23$ e Å$^{-3}$
Extinction correction: none
Scattering factors from International Tables Vol C 4.2.6.8 and 6.1.1.4

Table 1. *Selected geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| F102—C102 | 1.354 (3) | C7—C8 | 1.518 (4) |
| F103—C103 | 1.356 (3) | C8—C9 | 1.515 (4) |
| F104—C104 | 1.352 (3) | C9—C13 | 1.518 (5) |
| F105—C105 | 1.346 (3) | C9—C14 | 1.511 (5) |
| F106—C106 | 1.356 (3) | C101—C102 | 1.390 (4) |
| F202—C202 | 1.360 (3) | C101—C106 | 1.383 (4) |
| F203—C203 | 1.345 (3) | C101—B1 | 1.656 (4) |
| F204—C204 | 1.343 (3) | C102—C103 | 1.382 (4) |
| F205—C205 | 1.349 (3) | C103—C104 | 1.363 (4) |
| F206—C206 | 1.355 (3) | C104—C105 | 1.373 (4) |
| F302—C302 | 1.358 (3) | C105—C106 | 1.373 (3) |
| F303—C303 | 1.353 (3) | C201—C202 | 1.387 (4) |
| F304—C304 | 1.345 (4) | C201—C206 | 1.394 (3) |
| F305—C305 | 1.347 (3) | C201—B1 | 1.662 (4) |
| F306—C306 | 1.365 (3) | C202—C203 | 1.380 (4) |
| F402—C402 | 1.353 (3) | C203—C204 | 1.375 (4) |
| F403—C403 | 1.342 (3) | C204—C205 | 1.374 (4) |
| F404—C404 | 1.343 (3) | C205—C206 | 1.379 (4) |
| F405—C405 | 1.354 (3) | C301—C302 | 1.388 (4) |
| F406—C406 | 1.358 (3) | C301—C306 | 1.391 (4) |
| O1—C4 | 1.214 (3) | C301—B1 | 1.659 (4) |
| O2—C1 | 1.282 (3) | C302—C303 | 1.374 (4) |
| O2—C9 | 1.520 (4) | C303—C304 | 1.371 (4) |
| C1—C2 | 1.455 (4) | C304—C305 | 1.378 (4) |
| C1—C6 | 1.447 (4) | C305—C306 | 1.374 (4) |
| C2—C3 | 1.355 (4) | C401—C402 | 1.388 (3) |
| C2—C10 | 1.499 (4) | C401—C406 | 1.386 (4) |
| C3—C4 | 1.488 (4) | C401—B1 | 1.653 (4) |
| C3—C11 | 1.493 (4) | C402—C403 | 1.388 (4) |
| C4—C5 | 1.491 (4) | C403—C404 | 1.370 (4) |
| C5—C6 | 1.355 (4) | C404—C405 | 1.365 (4) |
| C5—C12 | 1.495 (4) | C405—C406 | 1.379 (4) |
| C6—C7 | 1.505 (4) | | |
| C1—O2—C9 | 123.5 (2) | C1—C2—C10 | 117.4 (2) |
| O2—C1—C2 | 113.2 (2) | C3—C2—C10 | 124.9 (3) |
| O2—C1—C6 | 121.8 (2) | C2—C3—C4 | 118.8 (3) |
| C2—C1—C6 | 125.0 (2) | C2—C3—C11 | 124.1 (3) |
| C1—C2—C3 | 117.7 (2) | C4—C3—C11 | 117.1 (2) |

| | | | |
|---|---|---|---|
| C3—C4—O1 | 119.9 (3) | F202—C202—C203 | 114.3 (2) |
| C3—C4—C5 | 120.6 (2) | C201—C202—C203 | 125.0 (2) |
| O1—C4—C5 | 119.5 (3) | F203—C203—C202 | 120.7 (2) |
| C4—C5—C6 | 119.4 (2) | F203—C203—C204 | 119.6 (3) |
| C4—C5—C12 | 117.7 (3) | C202—C203—C204 | 119.7 (3) |
| C6—C5—C12 | 122.9 (3) | F204—C204—C203 | 120.7 (3) |
| C1—C6—C5 | 117.2 (3) | F204—C204—C205 | 120.8 (3) |
| C1—C6—C7 | 119.0 (2) | C203—C204—C205 | 118.5 (3) |
| C5—C6—C7 | 123.7 (2) | F205—C205—C204 | 119.7 (3) |
| C6—C7—C8 | 110.9 (2) | F205—C205—C206 | 120.6 (3) |
| C7—C8—C9 | 111.4 (2) | C204—C205—C206 | 119.7 (3) |
| O2—C9—C8 | 107.2 (2) | F206—C206—C201 | 119.2 (2) |
| O2—C9—C13 | 102.3 (3) | F206—C206—C205 | 115.9 (2) |
| C8—C9—C13 | 112.8 (3) | C201—C206—C205 | 124.9 (3) |
| O2—C9—C14 | 106.0 (2) | C302—C301—C306 | 112.4 (2) |
| C8—C9—C14 | 114.0 (3) | C302—C301—B1 | 127.9 (2) |
| C13—C9—C14 | 113.3 (3) | C306—C301—B1 | 119.3 (2) |
| C102—C101—C106 | 112.9 (2) | F302—C302—C301 | 120.3 (2) |
| C102—C101—B1 | 127.9 (2) | F302—C302—C303 | 115.3 (2) |
| C106—C101—B1 | 118.9 (2) | C301—C302—C303 | 124.4 (2) |
| F102—C102—C101 | 121.1 (2) | F303—C303—C302 | 120.7 (2) |
| F102—C102—C103 | 115.3 (2) | F303—C303—C304 | 118.9 (3) |
| C101—C102—C103 | 123.6 (2) | C302—C303—C304 | 120.4 (2) |
| F103—C103—C102 | 120.3 (2) | F304—C304—C303 | 121.0 (3) |
| F103—C103—C104 | 119.4 (2) | F304—C304—C305 | 120.7 (3) |
| C102—C103—C104 | 120.3 (2) | C303—C304—C305 | 118.3 (3) |
| F104—C104—C103 | 120.8 (2) | F305—C305—C304 | 119.9 (3) |
| F104—C104—C105 | 120.4 (2) | F305—C305—C306 | 120.9 (3) |
| C103—C104—C105 | 118.8 (2) | C304—C305—C306 | 119.2 (2) |
| F105—C105—C104 | 120.0 (2) | F306—C306—C301 | 119.0 (2) |
| F105—C105—C106 | 120.9 (2) | F306—C306—C305 | 115.6 (2) |
| C104—C105—C106 | 119.0 (2) | C301—C306—C305 | 125.3 (2) |
| F106—C106—C101 | 119.2 (2) | C402—C401—C406 | 113.4 (2) |
| F106—C106—C105 | 115.5 (2) | C402—C401—B1 | 126.3 (2) |
| C101—C106—C105 | 125.3 (2) | C406—C401—B1 | 119.9 (2) |
| C202—C201—C206 | 112.3 (2) | F402—C402—C401 | 121.2 (2) |
| C202—C201—B1 | 127.3 (2) | F402—C402—C403 | 114.7 (2) |
| C206—C201—B1 | 119.8 (2) | C401—C402—C403 | 124.0 (2) |
| F202—C202—C201 | 120.7 (2) | C402—C403—F403 | 120.5 (2) |

| | | | |
|---|---|---|---|
| C402—C403—C404 | 119.5 (2) | F406—C406—C405 | 116.3 (2) |
| F403—C403—C404 | 120.0 (2) | C401—C406—C405 | 124.2 (2) |
| F404—C404—C403 | 120.4 (3) | C201—B1—C301 | 115.6 (2) |
| F404—C404—C405 | 120.7 (3) | C201—B1—C101 | 112.1 (2) |
| C403—C404—C405 | 118.9 (2) | C301—B1—C101 | 100.54 (19) |
| F405—C405—C404 | 120.1 (3) | C201—B1—C401 | 101.34 (19) |
| F405—C405—C406 | 120.0 (3) | C301—B1—C401 | 112.8 (2) |
| C404—C405—C406 | 120.0 (3) | C101—B1—C401 | 115.0 (2) |
| F406—C406—C401 | 119.5 (2) | | |

H atoms were included at calculated positions with methyl H atoms oriented to best-match peaks in difference electron density maps. Restraints were imposed on distances and angles involving H atoms as they were refined positionally.

Data collection: *COLLECT* (Nonius BV, 1997). Cell refinement: Denzo/Scalepack . Data reduction: Denzo/Scalepack (Otwinowski & Minor, 1997). Program(s) used to solve structure: *SIR*92 (Altomare *et al.* 1994). Program(s) used to refine structure: *CRYSTALS* (Watkin *et al.*2003). Molecular graphics: *ORTEP*–II (Johnson 1976) in teXsan (MSC, 1992–1997) . Software used to prepare material for publication: *CRYSTALS* .

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## Supplementary data

Table *S*1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$$U_{\mathrm{eq}} = (1/3)\Sigma_i\Sigma_j U^{ij} a^i a^j \mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $U_{\mathrm{eq}}$ |
|---|---|---|---|---|
| F102 | 0.09321 (14) | 0.66485 (9) | 0.33427 (9) | 0.0489 |
| F103 | −0.00144 (16) | 0.64108 (11) | 0.18820 (10) | 0.0600 |
| F104 | 0.07215 (15) | 0.52693 (11) | 0.10173 (9) | 0.0562 |
| F105 | 0.24729 (15) | 0.43652 (10) | 0.16726 (9) | 0.0517 |
| F106 | 0.34485 (13) | 0.45922 (9) | 0.31330 (9) | 0.0459 |
| F202 | 0.15873 (14) | 0.42113 (9) | 0.39293 (10) | 0.0513 |
| F203 | 0.16846 (17) | 0.30442 (10) | 0.48916 (13) | 0.0665 |
| F204 | 0.31250 (18) | 0.30448 (12) | 0.62722 (12) | 0.0711 |
| F205 | 0.44988 (16) | 0.42782 (12) | 0.66491 (10) | 0.0619 |
| F206 | 0.44446 (13) | 0.54608 (9) | 0.56820 (9) | 0.0449 |
| F302 | 0.53120 (13) | 0.49623 (8) | 0.43591 (9) | 0.0426 |
| F303 | 0.71073 (15) | 0.55768 (11) | 0.38770 (12) | 0.0573 |
| F304 | 0.70118 (17) | 0.69944 (12) | 0.32283 (13) | 0.0717 |
| F305 | 0.50263 (18) | 0.77965 (11) | 0.30803 (12) | 0.0666 |
| F306 | 0.32140 (14) | 0.72061 (9) | 0.35751 (10) | 0.0511 |
| F402 | 0.39924 (13) | 0.70280 (9) | 0.52790 (10) | 0.0453 |
| F403 | 0.29747 (15) | 0.77788 (9) | 0.62940 (9) | 0.0511 |
| F404 | 0.07569 (17) | 0.75258 (12) | 0.63935 (11) | 0.0670 |
| F405 | −0.04063 (15) | 0.64391 (13) | 0.54679 (11) | 0.0648 |
| F406 | 0.06188 (13) | 0.56186 (9) | 0.44904 (10) | 0.0487 |
| O1 | 0.1703 (2) | 0.86258 (13) | 0.49917 (12) | 0.0582 |
| O2 | 0.16612 (16) | 0.90667 (12) | 0.20085 (10) | 0.0466 |
| C1 | 0.1780 (2) | 0.88864 (15) | 0.27335 (14) | 0.0372 |
| C2 | 0.0786 (2) | 0.85155 (15) | 0.29534 (15) | 0.0385 |
| C3 | 0.0756 (2) | 0.84090 (15) | 0.37242 (16) | 0.0411 |
| C4 | 0.1756 (3) | 0.86386 (15) | 0.42978 (15) | 0.0430 |
| C5 | 0.2819 (2) | 0.88997 (15) | 0.40274 (16) | 0.0429 |
| C6 | 0.2816 (2) | 0.90412 (14) | 0.32586 (15) | 0.0407 |
| C7 | 0.3813 (2) | 0.93645 (18) | 0.29320 (18) | 0.0487 |
| C8 | 0.3702 (2) | 0.9219 (2) | 0.20604 (18) | 0.0525 |
| C9 | 0.2563 (2) | 0.9481 (2) | 0.16383 (18) | 0.0532 |
| C10 | −0.0147 (3) | 0.8304 (2) | 0.23097 (17) | 0.0509 |
| C11 | −0.0239 (3) | 0.80941 (19) | 0.40453 (19) | 0.0529 |
| C12 | 0.3835 (3) | 0.9024 (2) | 0.46325 (19) | 0.0603 |
| C13 | 0.2328 (3) | 0.9205 (3) | 0.07988 (19) | 0.0772 |
| C14 | 0.2339 (3) | 1.0324 (2) | 0.1725 (2) | 0.0722 |
| C101 | 0.2226 (2) | 0.56081 (14) | 0.33385 (14) | 0.0357 |
| C102 | 0.1347 (2) | 0.60581 (15) | 0.29679 (15) | 0.0390 |
| C103 | 0.0848 (2) | 0.59524 (16) | 0.22024 (16) | 0.0430 |
| C104 | 0.1214 (2) | 0.53847 (16) | 0.17635 (14) | 0.0417 |
| C105 | 0.2093 (2) | 0.49285 (15) | 0.20934 (15) | 0.0400 |
| C106 | 0.2572 (2) | 0.50571 (14) | 0.28538 (14) | 0.0366 |
| C201 | 0.3040 (2) | 0.49059 (14) | 0.47334 (14) | 0.0365 |
| C202 | 0.2361 (2) | 0.42634 (15) | 0.45893 (15) | 0.0404 |
| C203 | 0.2382 (2) | 0.36406 (16) | 0.50803 (18) | 0.0470 |
| C204 | 0.3106 (3) | 0.36377 (17) | 0.57779 (18) | 0.0503 |
| C205 | 0.3803 (2) | 0.42569 (18) | 0.59590 (16) | 0.0463 |
| C206 | 0.3752 (2) | 0.48646 (15) | 0.54484 (15) | 0.0375 |
| C301 | 0.4158 (2) | 0.60562 (14) | 0.40153 (14) | 0.0345 |
| C302 | 0.5186 (2) | 0.56774 (14) | 0.40543 (15) | 0.0372 |
| C303 | 0.6129 (2) | 0.59783 (17) | 0.38037 (16) | 0.0438 |
| C304 | 0.6095 (3) | 0.66930 (18) | 0.34761 (18) | 0.0495 |
| C305 | 0.5093 (3) | 0.70947 (16) | 0.34036 (17) | 0.0473 |
| C306 | 0.4175 (2) | 0.67730 (15) | 0.36700 (15) | 0.0397 |
| C401 | 0.2351 (2) | 0.62884 (14) | 0.48065 (14) | 0.0348 |
| C402 | 0.2896 (2) | 0.68460 (14) | 0.52952 (14) | 0.0368 |
| C403 | 0.2388 (2) | 0.72523 (15) | 0.58359 (15) | 0.0400 |

| | | | | |
|---|---|---|---|---|
| C404 | 0.1274 (3) | 0.71208 (17) | 0.58922 (15) | 0.0450 |
| C405 | 0.0698 (2) | 0.65762 (18) | 0.54272 (16) | 0.0446 |
| C406 | 0.1238 (2) | 0.61703 (15) | 0.49099 (15) | 0.0391 |
| B1 | 0.2946 (2) | 0.57163 (16) | 0.42271 (16) | 0.0343 |
| H71 | 0.383 (2) | 0.9919 (11) | 0.3035 (13) | 0.0557 |
| H72 | 0.4504 (16) | 0.9130 (13) | 0.3192 (12) | 0.0557 |
| H81 | 0.4287 (17) | 0.9500 (13) | 0.1851 (12) | 0.0600 |
| H82 | 0.380 (2) | 0.8668 (11) | 0.1971 (13) | 0.0600 |
| H101 | −0.0728 (18) | 0.8018 (14) | 0.2500 (13) | 0.0585 |
| H102 | −0.0467 (19) | 0.8758 (11) | 0.2062 (13) | 0.0585 |
| H103 | 0.0166 (19) | 0.8011 (13) | 0.1922 (13) | 0.0585 |
| H111 | −0.031 (2) | 0.8322 (14) | 0.4528 (12) | 0.0622 |
| H112 | −0.0905 (17) | 0.8168 (15) | 0.3693 (12) | 0.0622 |
| H113 | −0.017 (2) | 0.7558 (11) | 0.4121 (15) | 0.0622 |
| H121 | 0.368 (2) | 0.8933 (15) | 0.5146 (11) | 0.0671 |
| H122 | 0.411 (2) | 0.9543 (11) | 0.4606 (14) | 0.0671 |
| H123 | 0.4423 (18) | 0.8693 (14) | 0.4537 (14) | 0.0671 |
| H131 | 0.285 (2) | 0.9460 (16) | 0.0499 (14) | 0.0909 |
| H132 | 0.1565 (18) | 0.9322 (17) | 0.0577 (14) | 0.0909 |
| H133 | 0.243 (3) | 0.8653 (11) | 0.0775 (15) | 0.0909 |
| H141 | 0.294 (2) | 1.0609 (13) | 0.1535 (17) | 0.0857 |
| H142 | 0.233 (3) | 1.0452 (14) | 0.2262 (12) | 0.0857 |
| H143 | 0.1621 (19) | 1.0467 (14) | 0.1412 (15) | 0.0857 |

Table *S*2. *Anisotropic displacement parameters* (Å$^2$)

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|
| F102 | 0.0559 (9) | 0.0415 (8) | 0.0452 (8) | 0.0186 (7) | −0.0051 (7) | −0.0065 (7) |
| F103 | 0.0590 (10) | 0.0620 (11) | 0.0510 (9) | 0.0230 (9) | −0.0171 (8) | 0.0016 (8) |
| F104 | 0.0618 (10) | 0.0662 (11) | 0.0344 (8) | 0.0031 (9) | −0.0130 (7) | −0.0045 (7) |
| F105 | 0.0615 (10) | 0.0514 (10) | 0.0400 (8) | 0.0082 (8) | −0.0001 (7) | −0.0151 (7) |
| F106 | 0.0491 (9) | 0.0423 (8) | 0.0422 (8) | 0.0161 (7) | −0.0063 (7) | −0.0068 (6) |
| F202 | 0.0520 (9) | 0.0427 (9) | 0.0531 (9) | −0.0095 (7) | −0.0111 (7) | −0.0015 (7) |
| F203 | 0.0691 (12) | 0.0438 (10) | 0.0834 (13) | −0.0174 (9) | 0.0015 (10) | 0.0075 (9) |
| F204 | 0.0753 (13) | 0.0606 (12) | 0.0765 (12) | 0.0012 (10) | 0.0088 (10) | 0.0347 (10) |
| F205 | 0.0578 (10) | 0.0787 (13) | 0.0442 (9) | 0.0039 (9) | −0.0081 (8) | 0.0190 (9) |
| F206 | 0.0444 (8) | 0.0471 (9) | 0.0389 (8) | −0.0035 (7) | −0.0067 (6) | −0.0030 (7) |
| F302 | 0.0458 (8) | 0.0326 (7) | 0.0484 (8) | 0.0090 (6) | 0.0033 (6) | 0.0025 (6) |
| F303 | 0.0452 (9) | 0.0589 (11) | 0.0701 (11) | 0.0138 (8) | 0.0158 (8) | 0.0029 (9) |
| F304 | 0.0622 (12) | 0.0694 (13) | 0.0904 (14) | −0.0034 (10) | 0.0341 (10) | 0.0152 (10) |
| F305 | 0.0789 (13) | 0.0448 (10) | 0.0794 (13) | 0.0045 (9) | 0.0228 (10) | 0.0213 (9) |
| F306 | 0.0530 (9) | 0.0375 (8) | 0.0620 (10) | 0.0120 (7) | 0.0063 (8) | 0.0084 (7) |
| F402 | 0.0405 (8) | 0.0457 (9) | 0.0478 (8) | −0.0027 (7) | 0.0009 (6) | −0.0111 (7) |
| F403 | 0.0672 (11) | 0.0430 (9) | 0.0386 (8) | 0.0064 (8) | −0.0065 (7) | −0.0115 (7) |
| F404 | 0.0744 (12) | 0.0795 (13) | 0.0506 (10) | 0.0173 (10) | 0.0210 (9) | −0.0137 (9) |
| F405 | 0.0459 (9) | 0.0855 (14) | 0.0664 (11) | −0.0035 (9) | 0.0199 (8) | −0.0003 (10) |
| F406 | 0.0426 (8) | 0.0473 (9) | 0.0538 (9) | −0.0088 (7) | −0.0001 (7) | −0.0050 (7) |
| O1 | 0.0840 (15) | 0.0538 (12) | 0.0355 (10) | 0.0130 (11) | 0.0046 (10) | 0.0055 (9) |
| O2 | 0.0397 (10) | 0.0619 (12) | 0.0367 (10) | 0.0092 (9) | 0.0010 (8) | 0.0083 (9) |
| C1 | 0.0430 (13) | 0.0344 (13) | 0.0336 (12) | 0.0097 (11) | 0.0034 (10) | 0.0011 (10) |
| C2 | 0.0417 (13) | 0.0340 (13) | 0.0383 (13) | 0.0045 (11) | 0.0017 (10) | −0.0015 (10) |
| C3 | 0.0498 (15) | 0.0298 (12) | 0.0438 (14) | 0.0089 (11) | 0.0075 (11) | 0.0020 (11) |
| C4 | 0.0598 (17) | 0.0316 (13) | 0.0359 (13) | 0.0118 (12) | 0.0018 (11) | 0.0013 (10) |
| C5 | 0.0523 (15) | 0.0336 (13) | 0.0395 (13) | 0.0060 (12) | −0.0042 (11) | −0.0043 (11) |
| C6 | 0.0462 (14) | 0.0325 (13) | 0.0400 (13) | 0.0068 (11) | −0.0039 (11) | −0.0011 (10) |
| C7 | 0.0408 (14) | 0.0499 (17) | 0.0526 (16) | 0.0021 (12) | −0.0022 (12) | 0.0007 (13) |
| C8 | 0.0391 (15) | 0.066 (2) | 0.0511 (16) | 0.0083 (14) | 0.0040 (12) | 0.0077 (14) |
| C9 | 0.0389 (14) | 0.078 (2) | 0.0445 (15) | 0.0075 (14) | 0.0114 (12) | 0.0190 (14) |
| C10 | 0.0467 (16) | 0.0561 (18) | 0.0466 (15) | −0.0039 (13) | −0.0032 (12) | −0.0037 (13) |
| C11 | 0.0590 (18) | 0.0485 (17) | 0.0536 (16) | 0.0095 (14) | 0.0158 (14) | 0.0081 (13) |
| C12 | 0.067 (2) | 0.064 (2) | 0.0438 (15) | −0.0006 (17) | −0.0124 (14) | −0.0023 (15) |
| C13 | 0.0493 (18) | 0.143 (4) | 0.0392 (16) | 0.020 (2) | 0.0047 (13) | 0.014 (2) |
| C14 | 0.063 (2) | 0.072 (2) | 0.085 (2) | 0.0172 (18) | 0.0208 (19) | 0.038 (2) |
| C101 | 0.0379 (13) | 0.0328 (12) | 0.0344 (12) | 0.0009 (10) | −0.0009 (10) | −0.0009 (10) |
| C102 | 0.0446 (14) | 0.0339 (13) | 0.0361 (12) | 0.0073 (11) | −0.0013 (10) | −0.0012 (10) |
| C103 | 0.0417 (14) | 0.0408 (14) | 0.0425 (14) | 0.0062 (11) | −0.0068 (11) | 0.0035 (11) |
| C104 | 0.0465 (15) | 0.0456 (15) | 0.0297 (12) | −0.0016 (12) | −0.0046 (10) | −0.0008 (10) |
| C105 | 0.0464 (14) | 0.0375 (13) | 0.0352 (12) | −0.0013 (11) | 0.0035 (10) | −0.0071 (11) |
| C106 | 0.0369 (13) | 0.0325 (12) | 0.0373 (12) | 0.0069 (10) | −0.0047 (10) | 0.0000 (10) |
| C201 | 0.0376 (13) | 0.0329 (12) | 0.0377 (13) | 0.0033 (10) | 0.0012 (10) | −0.0020 (10) |
| C202 | 0.0396 (13) | 0.0382 (14) | 0.0410 (13) | 0.0020 (11) | −0.0013 (11) | −0.0025 (11) |
| C203 | 0.0453 (15) | 0.0355 (14) | 0.0601 (17) | −0.0019 (12) | 0.0082 (13) | 0.0036 (13) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C204 | 0.0508 (16) | 0.0451 (16) | 0.0556 (17) | 0.0057 (13) | 0.0096 (13) | 0.0178 (13) |
| C205 | 0.0417 (14) | 0.0557 (17) | 0.0403 (14) | 0.0087 (13) | 0.0024 (11) | 0.0075 (12) |
| C206 | 0.0366 (12) | 0.0349 (13) | 0.0397 (13) | 0.0024 (10) | 0.0012 (10) | −0.0010 (10) |
| C301 | 0.0394 (13) | 0.0298 (12) | 0.0325 (11) | 0.0033 (10) | 0.0000 (10) | −0.0033 (9) |
| C302 | 0.0461 (14) | 0.0308 (13) | 0.0331 (12) | 0.0024 (11) | 0.0015 (10) | −0.0010 (9) |
| C303 | 0.0429 (15) | 0.0446 (15) | 0.0445 (14) | 0.0085 (12) | 0.0081 (12) | −0.0036 (12) |
| C304 | 0.0507 (16) | 0.0484 (17) | 0.0521 (16) | −0.0038 (13) | 0.0161 (13) | −0.0001 (13) |
| C305 | 0.0606 (17) | 0.0341 (14) | 0.0477 (15) | 0.0039 (13) | 0.0104 (13) | 0.0061 (12) |
| C306 | 0.0422 (14) | 0.0358 (14) | 0.0399 (13) | 0.0071 (11) | 0.0026 (11) | −0.0009 (10) |
| C401 | 0.0373 (13) | 0.0309 (12) | 0.0341 (11) | 0.0044 (10) | −0.0012 (10) | −0.0003 (10) |
| C402 | 0.0377 (13) | 0.0356 (13) | 0.0352 (12) | 0.0025 (11) | −0.0007 (10) | 0.0016 (10) |
| C403 | 0.0519 (15) | 0.0350 (13) | 0.0306 (11) | 0.0022 (11) | −0.0018 (10) | −0.0003 (10) |
| C404 | 0.0553 (16) | 0.0480 (16) | 0.0327 (12) | 0.0127 (13) | 0.0096 (11) | 0.0011 (11) |
| C405 | 0.0400 (14) | 0.0548 (16) | 0.0397 (14) | 0.0036 (12) | 0.0082 (11) | 0.0083 (12) |
| C406 | 0.0426 (14) | 0.0370 (13) | 0.0356 (12) | −0.0003 (11) | −0.0003 (10) | −0.0005 (10) |
| B1 | 0.0364 (14) | 0.0311 (14) | 0.0329 (13) | 0.0036 (11) | −0.0029 (11) | −0.0020 (11) |

Table *S*3. *Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| F102—C102 | 1.354 (3) | C10—H102 | 0.954 (18) |
| F103—C103 | 1.356 (3) | C10—H103 | 0.965 (18) |
| F104—C104 | 1.352 (3) | C11—H111 | 0.943 (18) |
| F105—C105 | 1.346 (3) | C11—H112 | 0.936 (18) |
| F106—C106 | 1.356 (3) | C11—H113 | 0.950 (18) |
| F202—C202 | 1.360 (3) | C12—H121 | 0.951 (18) |
| F203—C203 | 1.345 (3) | C12—H122 | 0.970 (18) |
| F204—C204 | 1.343 (3) | C12—H123 | 0.944 (18) |
| F205—C205 | 1.349 (3) | C13—H131 | 0.980 (18) |
| F206—C206 | 1.355 (3) | C13—H132 | 0.957 (18) |
| F302—C302 | 1.358 (3) | C13—H133 | 0.975 (18) |
| F303—C303 | 1.353 (3) | C14—H141 | 0.969 (18) |
| F304—C304 | 1.345 (4) | C14—H142 | 0.960 (18) |
| F305—C305 | 1.347 (3) | C14—H143 | 0.975 (18) |
| F306—C306 | 1.365 (3) | C101—C102 | 1.390 (4) |
| F402—C402 | 1.353 (3) | C101—C106 | 1.383 (4) |
| F403—C403 | 1.342 (3) | C101—B1 | 1.656 (4) |
| F404—C404 | 1.343 (3) | C102—C103 | 1.382 (4) |
| F405—C405 | 1.354 (3) | C103—C104 | 1.363 (4) |
| F406—C406 | 1.358 (3) | C104—C105 | 1.373 (4) |
| O1—C4 | 1.214 (3) | C105—C106 | 1.373 (3) |
| O2—C1 | 1.282 (3) | C201—C202 | 1.387 (4) |
| O2—C9 | 1.520 (4) | C201—C206 | 1.394 (3) |
| C1—C2 | 1.455 (4) | C201—B1 | 1.662 (4) |
| C1—C6 | 1.447 (4) | C202—C203 | 1.380 (4) |
| C2—C3 | 1.355 (4) | C203—C204 | 1.375 (4) |
| C2—C10 | 1.499 (4) | C204—C205 | 1.374 (4) |
| C3—C4 | 1.488 (4) | C205—C206 | 1.379 (4) |
| C3—C11 | 1.493 (4) | C301—C302 | 1.388 (4) |
| C4—C5 | 1.491 (4) | C301—C306 | 1.391 (4) |
| C5—C6 | 1.355 (4) | C301—B1 | 1.659 (4) |
| C5—C12 | 1.495 (4) | C302—C303 | 1.374 (4) |
| C6—C7 | 1.505 (4) | C303—C304 | 1.371 (4) |
| C7—C8 | 1.518 (4) | C304—C305 | 1.378 (4) |
| C7—H71 | 0.986 (18) | C305—C306 | 1.374 (4) |
| C7—H72 | 0.971 (17) | C401—C402 | 1.388 (3) |
| C8—C9 | 1.515 (4) | C401—C406 | 1.386 (4) |
| C8—H81 | 0.968 (18) | C401—B1 | 1.653 (4) |
| C8—H82 | 0.986 (18) | C402—C403 | 1.388 (4) |
| C9—C13 | 1.518 (5) | C403—C404 | 1.370 (4) |
| C9—C14 | 1.511 (5) | C404—C405 | 1.365 (4) |
| C10—H101 | 0.955 (18) | C405—C406 | 1.379 (4) |
| C1—O2—C9 | 123.5 (2) | C2—C3—C4 | 118.8 (3) |
| O2—C1—C2 | 113.2 (2) | C2—C3—C11 | 124.1 (3) |
| O2—C1—C6 | 121.8 (2) | C4—C3—C11 | 117.1 (2) |
| C2—C1—C6 | 125.0 (2) | C3—C4—O1 | 119.9 (3) |
| C1—C2—C3 | 117.7 (2) | C3—C4—C5 | 120.6 (2) |
| C1—C2—C10 | 117.4 (2) | O1—C4—C5 | 119.5 (3) |
| C3—C2—C10 | 124.9 (3) | C4—C5—C6 | 119.4 (2) |

| | | | |
|---|---|---|---|
| C4—C5—C12 | 117.7 (3) | F105—C105—C106 | 120.9 (2) |
| C6—C5—C12 | 122.9 (3) | C104—C105—C106 | 119.0 (2) |
| C1—C6—C5 | 117.2 (3) | F106—C106—C101 | 119.2 (2) |
| C1—C6—C7 | 119.0 (2) | F106—C106—C105 | 115.5 (2) |
| C5—C6—C7 | 123.7 (2) | C101—C106—C105 | 125.3 (2) |
| C6—C7—C8 | 110.9 (2) | C202—C201—C206 | 112.3 (2) |
| C6—C7—H71 | 107.2 (13) | C202—C201—B1 | 127.3 (2) |
| C8—C7—H71 | 110.0 (13) | C206—C201—B1 | 119.8 (2) |
| C6—C7—H72 | 109.4 (13) | F202—C202—C201 | 120.7 (2) |
| C8—C7—H72 | 109.3 (13) | F202—C202—C203 | 114.3 (2) |
| H71—C7—H72 | 110.0 (15) | C201—C202—C203 | 125.0 (2) |
| C7—C8—C9 | 111.4 (2) | F203—C203—C202 | 120.7 (2) |
| C7—C8—H81 | 109.2 (13) | F203—C203—C204 | 119.6 (3) |
| C9—C8—H81 | 108.2 (13) | C202—C203—C204 | 119.7 (3) |
| C7—C8—H82 | 109.0 (13) | F204—C204—C203 | 120.7 (3) |
| C9—C8—H82 | 109.7 (13) | F204—C204—C205 | 120.8 (3) |
| H81—C8—H82 | 109.3 (15) | C203—C204—C205 | 118.5 (3) |
| O2—C9—C8 | 107.2 (2) | F205—C205—C204 | 119.7 (3) |
| O2—C9—C13 | 102.3 (3) | F205—C205—C206 | 120.6 (3) |
| C8—C9—C13 | 112.8 (3) | C204—C205—C206 | 119.7 (3) |
| O2—C9—C14 | 106.0 (2) | F206—C206—C201 | 119.2 (2) |
| C8—C9—C14 | 114.0 (3) | F206—C206—C205 | 115.9 (2) |
| C13—C9—C14 | 113.3 (3) | C201—C206—C205 | 124.9 (3) |
| C2—C10—H101 | 111.8 (14) | C302—C301—C306 | 112.4 (2) |
| C2—C10—H102 | 109.4 (14) | C302—C301—B1 | 127.9 (2) |
| H101—C10—H102 | 109.2 (15) | C306—C301—B1 | 119.3 (2) |
| C2—C10—H103 | 109.0 (14) | F302—C302—C301 | 120.3 (2) |
| H101—C10—H103 | 109.9 (16) | F302—C302—C303 | 115.3 (2) |
| H102—C10—H103 | 107.5 (16) | C301—C302—C303 | 124.4 (2) |
| C3—C11—H111 | 111.1 (14) | F303—C303—C302 | 120.7 (2) |
| C3—C11—H112 | 110.6 (14) | F303—C303—C304 | 118.9 (3) |
| H111—C11—H112 | 109.8 (16) | C302—C303—C304 | 120.4 (2) |
| C3—C11—H113 | 110.8 (14) | F304—C304—C303 | 121.0 (3) |
| H111—C11—H113 | 108.3 (16) | F304—C304—C305 | 120.7 (3) |
| H112—C11—H113 | 106.1 (16) | C303—C304—C305 | 118.3 (3) |
| C5—C12—H121 | 112.1 (14) | F305—C305—C304 | 119.9 (3) |
| C5—C12—H122 | 110.5 (14) | F305—C305—C306 | 120.9 (3) |
| H121—C12—H122 | 108.5 (16) | C304—C305—C306 | 119.2 (2) |
| C5—C12—H123 | 109.6 (14) | F306—C306—C301 | 119.0 (2) |
| H121—C12—H123 | 108.6 (16) | F306—C306—C305 | 115.6 (2) |
| H122—C12—H123 | 107.4 (16) | C301—C306—C305 | 125.3 (2) |
| C9—C13—H131 | 108.9 (15) | C402—C401—C406 | 113.4 (2) |
| C9—C13—H132 | 110.0 (15) | C402—C401—B1 | 126.3 (2) |
| H131—C13—H132 | 109.7 (16) | C406—C401—B1 | 119.9 (2) |
| C9—C13—H133 | 110.4 (15) | F402—C402—C401 | 121.2 (2) |
| H131—C13—H133 | 109.7 (16) | F402—C402—C403 | 114.7 (2) |
| H132—C13—H133 | 108.0 (17) | C401—C402—C403 | 124.0 (2) |
| C9—C14—H141 | 108.5 (15) | C402—C403—F403 | 120.5 (2) |
| C9—C14—H142 | 110.6 (15) | C402—C403—C404 | 119.5 (2) |
| H141—C14—H142 | 109.1 (16) | F403—C403—C404 | 120.0 (2) |
| C9—C14—H143 | 110.4 (15) | F404—C404—C403 | 120.4 (3) |
| H141—C14—H143 | 108.2 (16) | F404—C404—C405 | 120.7 (3) |
| H142—C14—H143 | 109.9 (16) | C403—C404—C405 | 118.9 (2) |
| C102—C101—C106 | 112.9 (2) | F405—C405—C404 | 120.1 (3) |
| C102—C101—B1 | 127.9 (2) | F405—C405—C406 | 120.0 (3) |
| C106—C101—B1 | 118.9 (2) | C404—C405—C406 | 120.0 (3) |
| F102—C102—C101 | 121.1 (2) | F406—C406—C401 | 119.5 (2) |
| F102—C102—C103 | 115.3 (2) | F406—C406—C405 | 116.3 (2) |
| C101—C102—C103 | 123.6 (2) | C401—C406—C405 | 124.2 (2) |
| F103—C103—C102 | 120.3 (2) | C201—B1—C301 | 115.6 (2) |
| F103—C103—C104 | 119.4 (2) | C201—B1—C101 | 112.1 (2) |
| C102—C103—C104 | 120.3 (2) | C301—B1—C101 | 100.54 (19) |
| F104—C104—C103 | 120.8 (2) | C201—B1—C401 | 101.34 (19) |
| F104—C104—C105 | 120.4 (2) | C301—B1—C401 | 112.8 (2) |
| C103—C104—C105 | 118.8 (2) | C101—B1—C401 | 115.0 (2) |
| F105—C105—C104 | 120.0 (2) | | |

| | | | |
|---|---|---|---|
| F102—C102—C101—C106 | −177.1 (2) | F102—C102—C103—F103 | −1.6 (4) |
| F102—C102—C101—B1 | −3.5 (4) | F102—C102—C103—C104 | 178.7 (2) |

| | |
|---|---|
| F103—C103—C102—C101 | 179.4 (2) |
| F103—C103—C104—F104 | −0.6 (4) |
| F103—C103—C104—C105 | 179.5 (2) |
| F104—C104—C103—C102 | 179.1 (2) |
| F104—C104—C105—F105 | 0.3 (4) |
| F104—C104—C105—C106 | −179.7 (2) |
| F105—C105—C104—C103 | −179.8 (2) |
| F105—C105—C106—F106 | 1.2 (4) |
| F105—C105—C106—C101 | −178.4 (2) |
| F106—C106—C101—C102 | 178.0 (2) |
| F106—C106—C101—B1 | 3.7 (3) |
| F106—C106—C105—C104 | −178.8 (2) |
| F202—C202—C201—C206 | 178.1 (2) |
| F202—C202—C201—B1 | 7.5 (4) |
| F202—C202—C203—F203 | 0.5 (4) |
| F202—C202—C203—C204 | −178.0 (3) |
| F203—C203—C202—C201 | 179.6 (3) |
| F203—C203—C204—F204 | 0.1 (5) |
| F203—C203—C204—C205 | −179.5 (3) |
| F204—C204—C203—C202 | 178.6 (3) |
| F204—C204—C205—F205 | −1.4 (5) |
| F204—C204—C205—C206 | −178.8 (3) |
| F205—C205—C204—C203 | 178.2 (3) |
| F205—C205—C206—F206 | 0.9 (4) |
| F205—C205—C206—C201 | −178.2 (2) |
| F206—C206—C201—C202 | −178.1 (2) |
| F206—C206—C201—B1 | −6.8 (4) |
| F206—C206—C205—C204 | 178.2 (3) |
| F302—C302—C301—C306 | −178.6 (2) |
| F302—C302—C301—B1 | −6.3 (4) |
| F302—C302—C303—F303 | −1.0 (4) |
| F302—C302—C303—C304 | 179.1 (2) |
| F303—C303—C302—C301 | 178.7 (2) |
| F303—C303—C304—F304 | 0.2 (4) |
| F303—C303—C304—C305 | 179.9 (3) |
| F304—C304—C303—C302 | −180.0 (3) |
| F304—C304—C305—F305 | 0.1 (4) |
| F304—C304—C305—C306 | −179.4 (3) |
| F305—C305—C304—C303 | −179.6 (3) |
| F305—C305—C306—F306 | 0.7 (4) |
| F305—C305—C306—C301 | −179.7 (2) |
| F306—C306—C301—C302 | 178.5 (2) |
| F306—C306—C301—B1 | 5.5 (3) |
| F306—C306—C305—C304 | −179.8 (2) |
| F402—C402—C401—C406 | 179.2 (2) |
| F402—C402—C401—B1 | 7.0 (4) |
| F402—C402—C403—F403 | −0.0 (3) |
| F402—C402—C403—C404 | 179.0 (2) |
| F403—C403—C402—C401 | 179.4 (2) |
| F403—C403—C404—F404 | 1.2 (4) |
| F403—C403—C404—C405 | −179.2 (2) |
| F404—C404—C403—C402 | −177.8 (2) |
| F404—C404—C405—F405 | 0.0 (4) |
| F404—C404—C405—C406 | 179.4 (2) |
| F405—C405—C404—C403 | −179.5 (2) |
| F405—C405—C406—F406 | −3.1 (4) |
| F405—C405—C406—C401 | 177.6 (2) |
| F406—C406—C401—C402 | −177.4 (2) |
| F406—C406—C401—B1 | −4.7 (3) |
| F406—C406—C405—C404 | 177.5 (2) |
| O1—C4—C3—C2 | 172.1 (3) |
| O1—C4—C3—C11 | −6.1 (4) |
| O1—C4—C5—C6 | −168.6 (3) |
| O1—C4—C5—C12 | 9.0 (4) |
| O2—C1—C2—C3 | −170.0 (2) |
| O2—C1—C2—C10 | 7.9 (4) |
| O2—C1—C6—C5 | 173.5 (2) |
| O2—C1—C6—C7 | −5.0 (4) |
| O2—C9—C8—C7 | −56.7 (3) |
| C1—O2—C9—C8 | 33.2 (4) |
| C1—O2—C9—C13 | 152.0 (3) |
| C1—O2—C9—C14 | −89.0 (3) |
| C1—C2—C3—C4 | −3.0 (4) |
| C1—C2—C3—C11 | 175.0 (3) |
| C1—C6—C5—C4 | −3.4 (4) |
| C1—C6—C5—C12 | 179.2 (3) |
| C1—C6—C7—C8 | −20.1 (3) |
| C2—C1—O2—C9 | 178.1 (2) |
| C2—C1—C6—C5 | −7.0 (4) |
| C2—C1—C6—C7 | 174.5 (3) |
| C2—C3—C4—C5 | −6.7 (4) |
| C3—C2—C1—C6 | 10.4 (4) |
| C3—C4—C5—C6 | 10.2 (4) |
| C3—C4—C5—C12 | −172.2 (3) |
| C4—C3—C2—C10 | 179.2 (3) |
| C4—C5—C6—C7 | 175.1 (2) |
| C5—C4—C3—C11 | 175.1 (3) |
| C5—C6—C7—C8 | 161.5 (3) |
| C6—C1—O2—C9 | −2.3 (4) |
| C6—C1—C2—C10 | −171.6 (3) |
| C6—C7—C8—C9 | 51.6 (3) |
| C7—C6—C5—C12 | −2.3 (4) |
| C7—C8—C9—C13 | −168.6 (3) |
| C7—C8—C9—C14 | 60.3 (4) |
| C10—C2—C3—C11 | −2.8 (4) |
| C101—C102—C103—C104 | −0.3 (4) |
| C101—C106—C105—C104 | 1.6 (4) |
| C101—B1—C201—C202 | −19.3 (4) |
| C101—B1—C201—C206 | 170.7 (2) |
| C101—B1—C301—C302 | −105.6 (3) |
| C101—B1—C301—C306 | 66.2 (3) |
| C101—B1—C401—C402 | −136.1 (2) |
| C101—B1—C401—C406 | 52.2 (3) |
| C102—C101—C106—C105 | −2.5 (4) |
| C102—C101—B1—C201 | 131.2 (3) |
| C102—C101—B1—C301 | −105.5 (3) |
| C102—C101—B1—C401 | 16.1 (4) |
| C102—C103—C104—C105 | −0.8 (4) |
| C103—C102—C101—C106 | 1.8 (4) |
| C103—C102—C101—B1 | 175.4 (2) |
| C103—C104—C105—C106 | 0.2 (4) |
| C105—C106—C101—B1 | −176.7 (2) |
| C106—C101—B1—C201 | −55.6 (3) |
| C106—C101—B1—C301 | 67.8 (3) |
| C106—C101—B1—C401 | −170.7 (2) |
| C201—C202—C203—C204 | 1.1 (4) |
| C201—C206—C205—C204 | −0.8 (4) |
| C201—B1—C301—C302 | 15.3 (4) |
| C201—B1—C301—C306 | −172.9 (2) |
| C201—B1—C401—C402 | 102.7 (3) |
| C201—B1—C401—C406 | −69.0 (3) |
| C202—C201—C206—C205 | 0.9 (4) |
| C202—C201—B1—C301 | −133.8 (3) |
| C202—C201—B1—C401 | 103.9 (3) |
| C202—C203—C204—C205 | −0.9 (5) |
| C203—C202—C201—C206 | −1.0 (4) |
| C203—C202—C201—B1 | −171.6 (3) |
| C203—C204—C205—C206 | 0.8 (5) |
| C205—C206—C201—B1 | 172.2 (2) |
| C206—C201—B1—C301 | 56.3 (3) |
| C206—C201—B1—C401 | −66.1 (3) |
| C301—C302—C303—C304 | −1.2 (4) |
| C301—C306—C305—C304 | −0.2 (4) |
| C301—B1—C401—C402 | −21.5 (3) |
| C301—B1—C401—C406 | 166.8 (2) |
| C302—C301—C306—C305 | −1.1 (4) |
| C302—C301—B1—C401 | 131.3 (3) |
| C302—C303—C304—C305 | −0.3 (4) |

| | | | |
|---|---|---|---|
| C303—C302—C301—C306 | 1.8 (4) | C402—C401—C406—C405 | 1.9 (4) |
| C303—C302—C301—B1 | 174.0 (2) | C402—C403—C404—C405 | 1.7 (4) |
| C303—C304—C305—C306 | 0.9 (4) | C403—C402—C401—C406 | −0.2 (4) |
| C305—C306—C301—B1 | −174.1 (2) | C403—C402—C401—B1 | −172.3 (2) |
| C306—C301—B1—C401 | −56.9 (3) | C403—C404—C405—C406 | −0.2 (4) |
| C401—C402—C403—C404 | −1.6 (4) | C405—C406—C401—B1 | 174.5 (2) |
| C401—C406—C405—C404 | −1.8 (4) | | |

Table *S*4. *Contact distances (Å)*

| | | | |
|---|---|---|---|
| F102···C3 | 3.164 (3) | F206···O2$^{vi}$ | 3.337 (2) |
| F102···C11 | 3.219 (4) | F206···C13$^{vi}$ | 3.472 (4) |
| F102···C2 | 3.335 (3) | F206···C10$^{vi}$ | 3.528 (4) |
| F102···C10 | 3.543 (4) | F302···C4$^{iii}$ | 2.900 (3) |
| F103···F403$^{i}$ | 2.843 (2) | F302···O1$^{iii}$ | 2.980 (3) |
| F103···C10 | 3.404 (4) | F302···C3$^{iii}$ | 3.010 (3) |
| F104···F406$^{ii}$ | 3.055 (2) | F302···C11$^{iii}$ | 3.362 (4) |
| F104···C12$^{i}$ | 3.275 (4) | F302···C13$^{vi}$ | 3.510 (4) |
| F104···C406$^{ii}$ | 3.282 (3) | F303···C5$^{iii}$ | 3.056 (3) |
| F104···C203$^{ii}$ | 3.346 (3) | F303···C6$^{iii}$ | 3.060 (3) |
| F104···F405$^{ii}$ | 3.357 (3) | F303···C13$^{vi}$ | 3.323 (4) |
| F104···C405$^{ii}$ | 3.386 (4) | F303···C4$^{iii}$ | 3.508 (3) |
| F104···C202$^{ii}$ | 3.480 (3) | F303···C7$^{iii}$ | 3.515 (4) |
| F104···C204$^{ii}$ | 3.510 (4) | F303···C12$^{iii}$ | 3.541 (4) |
| F105···C206$^{ii}$ | 3.106 (3) | F303···C1$^{iii}$ | 3.549 (3) |
| F105···F206$^{ii}$ | 3.135 (2) | F304···F404$^{vii}$ | 3.408 (3) |
| F105···C404$^{ii}$ | 3.171 (3) | F305···C7 | 3.096 (4) |
| F105···C403$^{ii}$ | 3.175 (3) | F305···F404$^{vii}$ | 3.226 (3) |
| F105···C205$^{ii}$ | 3.236 (4) | F305···C8 | 3.312 (4) |
| F105···C405$^{ii}$ | 3.237 (3) | F305···C6 | 3.474 (3) |
| F105···C402$^{ii}$ | 3.288 (3) | F306···C5 | 3.120 (3) |
| F105···C406$^{ii}$ | 3.319 (3) | F306···C6 | 3.280 (3) |
| F105···F205$^{ii}$ | 3.396 (3) | F306···C4 | 3.399 (4) |
| F105···C401$^{ii}$ | 3.412 (3) | F306···C1 | 3.598 (3) |
| F105···C10$^{iii}$ | 3.436 (4) | F402···C10$^{vi}$ | 3.560 (3) |
| F106···C10$^{iii}$ | 3.266 (4) | F403···O1 | 2.925 (3) |
| F106···C11$^{iii}$ | 3.328 (4) | F403···C10$^{vi}$ | 3.244 (4) |
| F106···C2$^{iii}$ | 3.424 (3) | F403···C14$^{viii}$ | 3.511 (4) |
| F106···C3$^{iii}$ | 3.476 (3) | F404···O1 | 3.424 (3) |
| F202···F305$^{iv}$ | 3.307 (2) | F405···C13$^{ix}$ | 3.068 (4) |
| F202···C7$^{iv}$ | 3.509 (3) | F405···C8$^{ix}$ | 3.313 (4) |
| F203···C305$^{iv}$ | 3.396 (4) | F406···C12$^{iv}$ | 3.543 (4) |
| F203···C304$^{iv}$ | 3.402 (4) | O1···C403 | 2.867 (3) |
| F203···F305$^{iv}$ | 3.475 (3) | O1···C404 | 3.143 (4) |
| F203···F304$^{iv}$ | 3.490 (3) | O1···C402 | 3.432 (3) |
| F204···F404$^{iii}$ | 3.251 (3) | O1···C14$^{viii}$ | 3.504 (4) |
| F205···C105$^{v}$ | 3.386 (3) | C4···C403 | 3.598 (4) |
| F205···C14$^{vi}$ | 3.449 (4) | C13···C303$^{i}$ | 3.550 (4) |
| F205···F404$^{iii}$ | 3.473 (3) | C104···C204$^{ii}$ | 3.484 (4) |
| F205···C106$^{v}$ | 3.533 (3) | C105···C205$^{ii}$ | 3.362 (4) |

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

## Table of Least-Squares Planes

---------- Plane number 1 ----------

| Atoms Defining Plane | | | Distance | esd |
|---|---|---|---|---|
| C1 | ( | 1) | -0.0608 | 0.0026 |
| C2 | ( | 1) | 0.0475 | 0.0026 |
| C3 | ( | 1) | 0.0118 | 0.0026 |
| C4 | ( | 1) | -0.0656 | 0.0028 |
| C5 | ( | 1) | 0.0470 | 0.0026 |
| C6 | ( | 1) | 0.0109 | 0.0025 |

| Additional Atoms | | | Distance |
|---|---|---|---|
| O1 | ( | 1) | -0.2436 |
| O2 | ( | 1) | -0.2217 |
| C7 | ( | 1) | 0.0081 |
| C8 | ( | 1) | 0.4153 |
| C9 | ( | 1) | -0.4028 |
| C10 | ( | 1) | 0.1428 |
| C11 | ( | 1) | 0.0034 |
| C12 | ( | 1) | 0.1392 |
| C13 | ( | 1) | 0.1532 |
| C14 | ( | 1) | -1.8884 |

Mean deviation from plane is 0.0406 angstroms
Chi-squared: 1791.2

---------- Plane number 2 ----------

| Atoms Defining Plane | | | Distance | esd |
|---|---|---|---|---|
| O2 | ( | 1) | 0.0037 | 0.0021 |
| C1 | ( | 1) | 0.0854 | 0.0026 |
| C6 | ( | 1) | -0.0352 | 0.0025 |
| C7 | ( | 1) | -0.1484 | 0.0031 |
| C8 | ( | 1) | 0.3663 | 0.0034 |
| C9 | ( | 1) | -0.2752 | 0.0034 |

| Additional Atoms | | | Distance |
|---|---|---|---|
| O1 | ( | 1) | -0.3001 |
| C2 | ( | 1) | 0.3164 |
| C3 | ( | 1) | 0.2129 |
| C4 | ( | 1) | -0.0605 |
| C5 | ( | 1) | -0.0695 |
| C10 | ( | 1) | 0.6052 |
| C11 | ( | 1) | 0.3147 |
| C12 | ( | 1) | -0.1738 |
| C13 | ( | 1) | 0.4069 |

Mean deviation from plane is 0.1524 angstroms
Chi-squared: 21205.3

Dihedral angles between least-squares planes

| plane | plane | angle |
|---|---|---|
| 2 | 1 | 7.65 |

Table of Least-Squares Planes (continued)

---------- Plane number 3 ----------

| Atoms Defining Plane | | | Distance | esd |
|---|---|---|---|---|
| O2 | ( | 1) | 0.0204 | 0.0021 |
| C1 | ( | 1) | 0.0056 | 0.0026 |
| C6 | ( | 1) | -0.0367 | 0.0025 |
| C7 | ( | 1) | 0.0510 | 0.0031 |
| C9 | ( | 1) | -0.0563 | 0.0034 |

| Additional Atoms | | | Distance |
|---|---|---|---|
| C8 | ( | 1) | 0.6543 |
| O1 | ( | 1) | -0.7123 |
| C2 | ( | 1) | 0.0461 |
| C3 | ( | 1) | -0.1729 |
| C4 | ( | 1) | -0.3726 |
| C5 | ( | 1) | -0.1850 |
| C10 | ( | 1) | 0.2807 |
| C11 | ( | 1) | -0.2703 |
| C12 | ( | 1) | -0.2268 |
| C13 | ( | 1) | 0.6826 |
| C14 | ( | 1) | -1.5271 |

Mean deviation from plane is 0.0340 angstroms
Chi-squared: 865.2

Dihedral angles between least-squares planes

| plane | plane | angle |
|---|---|---|
| 3 | 1 | 7.88 |
| 3 | 2 | 7.68 |

**Figure Captions for $(C_{14}H_{19}O_2)^+(B(C_6F_5)_4)^-$**

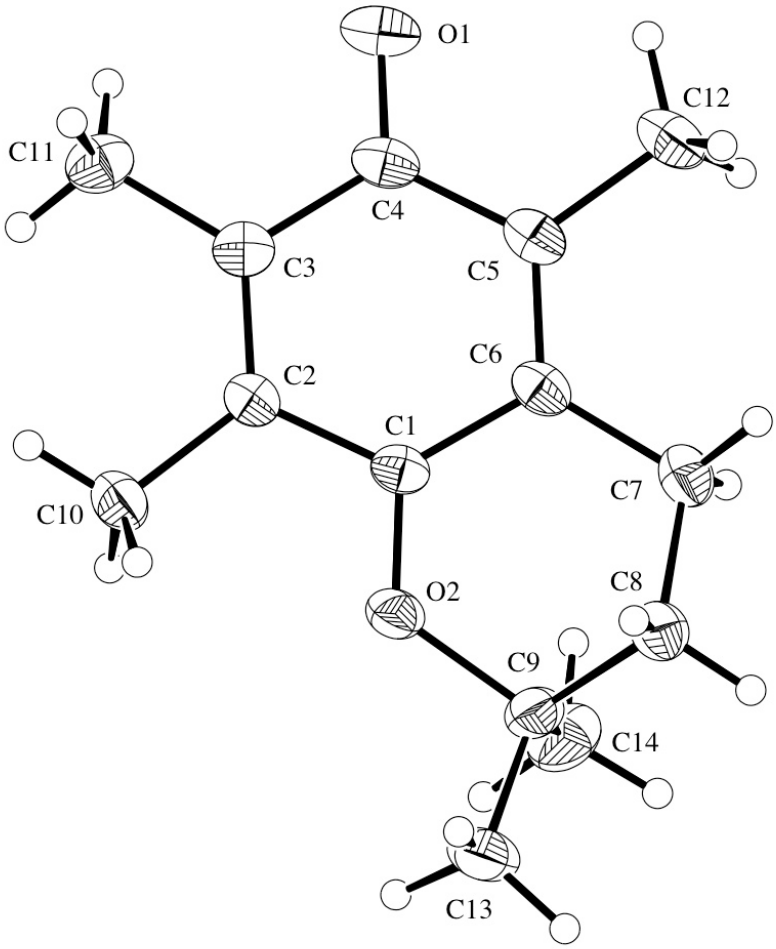


**Figure 1**. Molecular structure of the cation of $(C_{14}H_{19}O_2)^+(B(C_6F_5)_4)^-$ with labelling of selected atoms. Anisotropic displacement ellipsoids show 30% probability levels. Hydrogen atoms are drawn as circles with small radii.

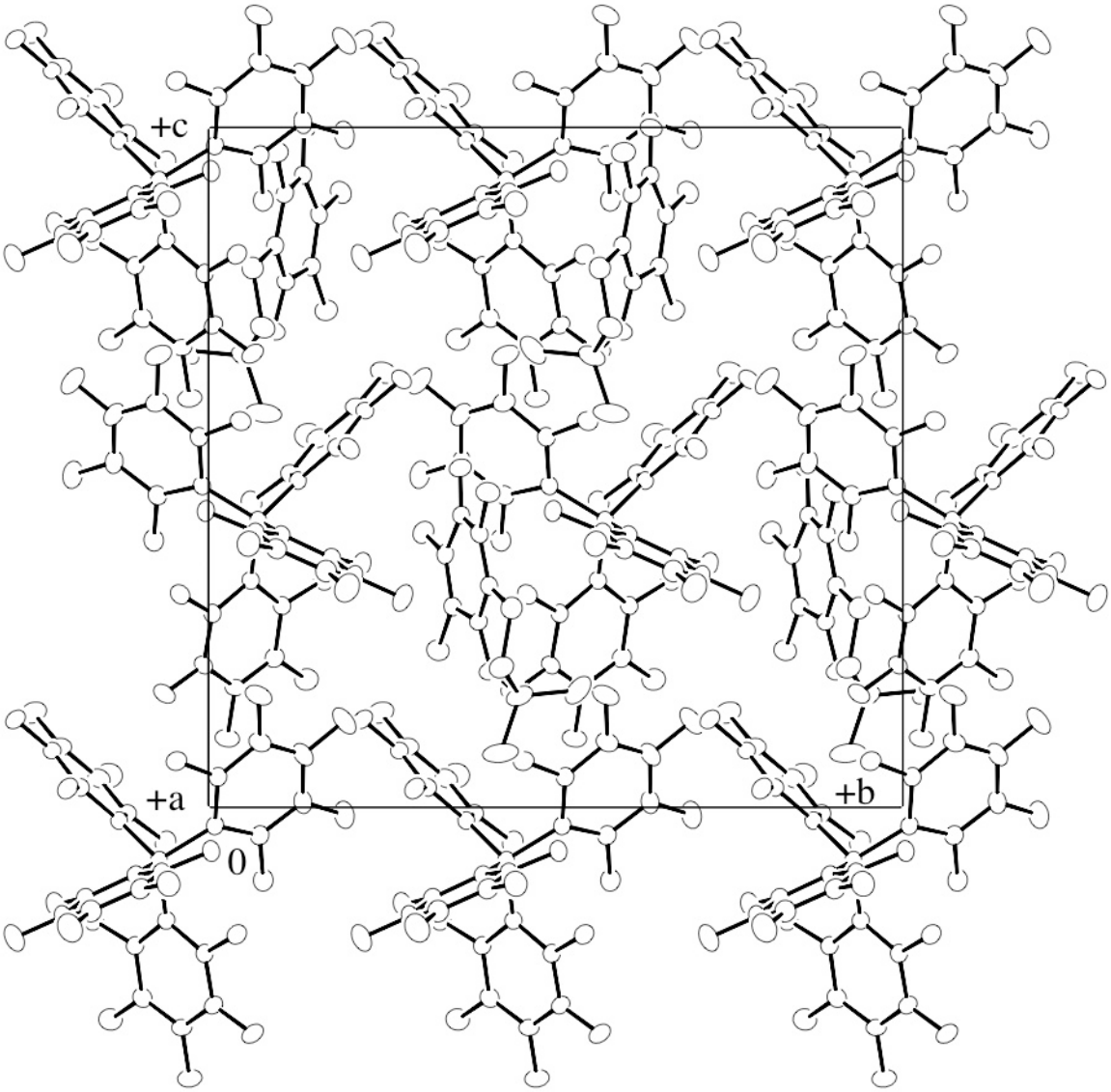


**Figure 2**. Unit cell packing diagram of $(C_{14}H_{19}O_2)^+(B(C_6F_5)_4)^-$ projected down the *a* axis. Hydrogen atoms are not shown.

9 May 2006

## Crystal structure of $C_{14}H_{20}O_2$ –web0603

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### Abstract

The crystal structure of $C_{14}H_{20}O_2$ is reported.

### Comment

The crystallographic asymmetric unit consists of two $C_{14}H_{20}O_2$ molecules.

The structure was solved by direct methods in space group P1, revealing 4 molecules in the unit cell. A centre of inversion was identified [*PLATON*]. Appropriate translations were applied and 2 molecules discarded as redundant. All further refinement was performed in space group $P\bar{1}$.

H atoms bonded to carbon were included at calculated positions with methyl H atoms oriented to best-match peaks in difference electron density maps. They were then refined positionally with restraints being imposed on distances and angles involving them. A difference electron density map at this stage revealed two alternative positions for each alcohol H atom. These were included in refinement with occupancies of 0.5 and refined positionally. A subsequent difference map revealed peaks in the methyl groups attached to $sp^2$ C atoms. Alternative H sites were therefore added between the original ones and the relative populations refined. These minor sites have not been refined positionally but simply ride on their respective C atoms.

There is hydrogen bonding between the alcohol groups of adjacent molecules giving rise to a chain extending in the a direction. There are two possible connectivities: O1[ $-x$,1-$y$,1-$z$]...H11[x,$y$,$z$]—O1[x,$y$,$z$]... H1012[1 $-x$,1-$y$,1-$z$]—O101[1 $-x$,1-$y$,1-$z$]...H1011[x,$y$,$z$]—O101[x,$y$,$z$]... H12[1 $-x$,1-$y$,1-$z$]—O1[1 $-x$,1-$y$,1-$z$] or O1[ $-x$,1-$y$,1-$z$]—H11[ $-x$,1-$y$,1-$z$]...O1[x,$y$,$z$]—H12[x,$y$,$z$]... O101[1 $-x$,1-$y$,1-$z$]—H1011[1 $-x$,1-$y$,1-$z$]... O101[x,$y$,$z$]—H1012[x,$y$,$z$]...O1[1 $-x$,1-$y$,1-$z$]. The structure reported here is the average of these.

The largest peaks in the final difference map are all of near-equal intensity and have no chemical significance.

## Experimental

The compound was prepared by SBL and recrystallized from methanol/water. The sample ID is SBL-06–4–26.

*Crystal data*

$C_{14}H_{20}O_2$
$M_r = 220.31$
Triclinic
$P\overline{1}$
$a = 9.0787\,(2)$ Å
$b = 10.7742\,(3)$ Å
$c = 13.2895\,(3)$ Å
$\alpha = 91.1222\,(16)°$
$\beta = 103.4637\,(14)°$
$\gamma = 97.1591\,(16)°$
$V = 1252.75\,(5)$ Å$^3$
$Z = 4$
$D_x = 1.168$ Mg m$^{-3}$
$D_m$ not measured
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 12916 reflections
$\theta = 3$–$27°$
$\mu = 0.076$ mm$^{-1}$
$T = 200$ K
Block
Colourless
0.48 × 0.22 × 0.21 mm
Crystal source: local

*Data collection*

Nonius KappaCCD diffractometer
$\varphi$ and $\omega$ scans with CCD
Absorption correction:
by integration *via* Gaussian method (Coppens, 1970) implemented in maXus (2000)
$T_{\min} = 0.973$, $T_{\max} = 0.989$
20668 measured reflections
5745 independent reflections

4234 reflections with
$I > 2.0\sigma(I)$
$R_{\rm int} = 0.034$
$\theta_{\max} = 27.553°$
$h = -11 \rightarrow 10$
$k = -14 \rightarrow 13$
$l = -17 \rightarrow 17$

*Refinement*

Refinement on $F^2$
$R(F) = 0.0382$
$wR(F^2) = 0.0982$
$S = 0.9001$
5732 reflections
421 parameters
H atoms treated by a mixture of independent and constrained refinement

Method= Modified Sheldrick $w$=1/[$\sigma^2(F^2)$ + ( 0.05$P$)$^2$ + 0.09$P$] ,
where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.007584$
$\Delta\rho_{\max} = 0.23$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.27$ e Å$^{-3}$
Extinction correction: none
Scattering factors from International Tables Vol C 4.2.6.8 and 6.1.1.4

Table 1. *Selected geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1—C4 | 1.3950 (13) | C8—C9 | 1.5217 (16) |
| O2—C1 | 1.3827 (12) | C9—C13 | 1.5159 (16) |
| O2—C9 | 1.4566 (12) | C9—C14 | 1.5207 (16) |
| O101—C104 | 1.3923 (13) | C101—C102 | 1.4012 (14) |
| O102—C101 | 1.3851 (12) | C101—C106 | 1.3926 (14) |
| O102—C109 | 1.4532 (12) | C102—C103 | 1.3963 (14) |
| C1—C2 | 1.4055 (14) | C102—C110 | 1.5019 (15) |
| C1—C6 | 1.3934 (14) | C103—C104 | 1.3979 (14) |
| C2—C3 | 1.3954 (14) | C103—C111 | 1.5074 (15) |
| C2—C10 | 1.5084 (14) | C104—C105 | 1.3884 (15) |
| C3—C4 | 1.3974 (15) | C105—C106 | 1.4023 (14) |
| C3—C11 | 1.5113 (15) | C105—C112 | 1.5086 (15) |
| C4—C5 | 1.3929 (15) | C106—C107 | 1.5112 (14) |
| C5—C6 | 1.4012 (15) | C107—C108 | 1.5212 (16) |
| C5—C12 | 1.5097 (15) | C108—C109 | 1.5233 (16) |
| C6—C7 | 1.5132 (14) | C109—C113 | 1.5172 (16) |
| C7—C8 | 1.5202 (16) | C109—C114 | 1.5200 (16) |

| | | | |
|---|---|---|---|
| C1—O2—C9 | 117.42 (8) | C13—C9—C14 | 110.49 (10) |
| C101—O102—C109 | 116.67 (8) | O102—C101—C102 | 115.28 (9) |
| O2—C1—C2 | 115.63 (9) | O102—C101—C106 | 122.66 (9) |
| O2—C1—C6 | 122.69 (9) | C102—C101—C106 | 122.06 (9) |
| C2—C1—C6 | 121.67 (9) | C101—C102—C103 | 118.98 (9) |
| C1—C2—C3 | 118.91 (9) | C101—C102—C110 | 119.91 (9) |
| C1—C2—C10 | 119.96 (9) | C103—C102—C110 | 121.11 (9) |
| C3—C2—C10 | 121.13 (9) | C102—C103—C104 | 118.67 (9) |
| C2—C3—C4 | 119.09 (9) | C102—C103—C111 | 121.23 (10) |
| C2—C3—C11 | 120.68 (10) | C104—C103—C111 | 120.10 (10) |
| C4—C3—C11 | 120.23 (10) | C103—C104—O101 | 118.70 (9) |
| C3—C4—O1 | 118.57 (9) | C103—C104—C105 | 122.47 (9) |
| C3—C4—C5 | 122.08 (10) | O101—C104—C105 | 118.83 (10) |
| O1—C4—C5 | 119.35 (9) | C104—C105—C106 | 118.91 (9) |
| C4—C5—C6 | 118.86 (9) | C104—C105—C112 | 120.74 (10) |
| C4—C5—C12 | 121.11 (10) | C106—C105—C112 | 120.35 (10) |
| C6—C5—C12 | 120.03 (10) | C105—C106—C101 | 118.85 (9) |
| C5—C6—C1 | 119.19 (9) | C105—C106—C107 | 120.80 (9) |
| C5—C6—C7 | 120.66 (9) | C101—C106—C107 | 120.35 (9) |
| C1—C6—C7 | 120.15 (9) | C106—C107—C108 | 111.43 (9) |
| C6—C7—C8 | 111.53 (9) | C107—C108—C109 | 111.27 (9) |
| C7—C8—C9 | 111.31 (9) | C108—C109—O102 | 107.72 (8) |
| C8—C9—O2 | 107.95 (9) | C108—C109—C113 | 111.45 (10) |
| C8—C9—C13 | 111.68 (10) | O102—C109—C113 | 104.83 (9) |
| O2—C9—C13 | 104.50 (9) | C108—C109—C114 | 113.33 (10) |
| C8—C9—C14 | 112.69 (9) | O102—C109—C114 | 108.67 (9) |
| O2—C9—C14 | 109.15 (9) | C113—C109—C114 | 110.42 (10) |

Table 2. *Hydrogen-bonding geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H11···O1$^{i}$ | 0.82 (2) | 1.90 (2) | 2.713 (2) | 171 (3) |
| O1—H12···O101$^{ii}$ | 0.82 (2) | 1.86 (2) | 2.683 (1) | 178 (3) |
| O101—H1012···O1$^{ii}$ | 0.81 (2) | 1.89 (2) | 2.683 (1) | 171 (3) |
| O101—H1011···O101$^{ii}$ | 0.81 (2) | 1.83 (3) | 2.634 (2) | 178 (3) |

Symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $1-x, 1-y, 1-z$.

H atoms were were refined positionally or ride on their respective C atom.

Data collection: *COLLECT* (Nonius BV, 1997). Cell refinement: Denzo/Scalepack . Data reduction: Denzo/Scalepack (Otwinowski & Minor, 1997). Program(s) used to solve structure: *SIR*92 (Altomare *et al.* 1994). Program(s) used to refine structure: *CRYSTALS* (Watkin *et al.*2003). Molecular graphics: *ORTEP*–II (Johnson 1976) in teXsan (MSC, 1992–1997) . Software used to prepare material for publication: *CRYSTALS* .

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## Supplementary data

Table *S*1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$$U_{\mathrm{eq}} = (1/3)\Sigma_i\Sigma_j U^{ij} a^i a^j \mathbf{a}_i.\mathbf{a}_j.$$

| | Occupancy | $x$ | $y$ | $z$ | $U_{\mathrm{eq}}$ |
|---|---|---|---|---|---|
| O1 | 1.0000 | 0.12791 (10) | 0.54457 (8) | 0.46899 (7) | 0.0441 |
| O2 | 1.0000 | 0.08699 (9) | 0.90889 (7) | 0.18008 (6) | 0.0348 |
| O101 | 1.0000 | 0.60224 (11) | 0.53152 (9) | 0.44595 (7) | 0.0461 |
| O102 | 1.0000 | 0.46750 (9) | 0.81266 (7) | 0.10154 (5) | 0.0338 |
| C1 | 1.0000 | 0.10484 (11) | 0.82045 (9) | 0.25445 (8) | 0.0287 |
| C2 | 1.0000 | 0.00232 (11) | 0.70908 (10) | 0.23093 (8) | 0.0287 |
| C3 | 1.0000 | 0.01365 (12) | 0.61527 (10) | 0.30218 (8) | 0.0300 |
| C4 | 1.0000 | 0.12226 (12) | 0.63680 (10) | 0.39648 (8) | 0.0307 |
| C5 | 1.0000 | 0.22402 (12) | 0.74707 (10) | 0.42001 (8) | 0.0314 |
| C6 | 1.0000 | 0.21838 (11) | 0.83851 (10) | 0.34617 (8) | 0.0297 |
| C7 | 1.0000 | 0.33517 (13) | 0.95479 (11) | 0.36466 (9) | 0.0362 |
| C8 | 1.0000 | 0.32783 (13) | 1.02695 (11) | 0.26684 (10) | 0.0383 |
| C9 | 1.0000 | 0.16336 (13) | 1.03566 (10) | 0.21056 (9) | 0.0345 |
| C10 | 1.0000 | −0.11654 (13) | 0.69213 (11) | 0.12962 (9) | 0.0352 |
| C11 | 1.0000 | −0.09070 (14) | 0.49229 (11) | 0.27848 (10) | 0.0394 |
| C12 | 1.0000 | 0.33926 (15) | 0.76876 (13) | 0.52289 (9) | 0.0429 |
| C13 | 1.0000 | 0.15451 (17) | 1.09984 (12) | 0.10924 (10) | 0.0466 |
| C14 | 1.0000 | 0.07666 (14) | 1.09927 (11) | 0.27733 (10) | 0.0396 |
| C101 | 1.0000 | 0.49406 (11) | 0.73868 (9) | 0.18630 (7) | 0.0272 |
| C102 | 1.0000 | 0.61672 (11) | 0.78680 (9) | 0.26820 (8) | 0.0286 |
| C103 | 1.0000 | 0.65170 (11) | 0.71786 (10) | 0.35652 (8) | 0.0298 |
| C104 | 1.0000 | 0.56504 (12) | 0.60172 (10) | 0.35896 (8) | 0.0306 |
| C105 | 1.0000 | 0.44292 (12) | 0.55410 (10) | 0.27816 (8) | 0.0301 |
| C106 | 1.0000 | 0.40484 (11) | 0.62492 (9) | 0.19069 (8) | 0.0281 |
| C107 | 1.0000 | 0.26957 (13) | 0.57898 (11) | 0.10245 (9) | 0.0341 |
| C108 | 1.0000 | 0.23195 (13) | 0.68080 (12) | 0.02654 (9) | 0.0361 |
| C109 | 1.0000 | 0.37633 (12) | 0.75150 (10) | 0.00465 (8) | 0.0332 |
| C110 | 1.0000 | 0.70892 (13) | 0.91026 (11) | 0.25963 (10) | 0.0370 |
| C111 | 1.0000 | 0.78058 (14) | 0.76620 (12) | 0.44772 (9) | 0.0403 |
| C112 | 1.0000 | 0.35284 (15) | 0.42800 (11) | 0.28378 (10) | 0.0404 |
| C113 | 1.0000 | 0.33998 (17) | 0.85761 (13) | −0.06655 (10) | 0.0471 |
| C114 | 1.0000 | 0.47228 (15) | 0.66733 (12) | −0.03806 (10) | 0.0396 |
| H11 | 0.5000 | 0.050 (2) | 0.511 (3) | 0.483 (2) | 0.0520 |
| H12 | 0.5000 | 0.210 (2) | 0.520 (3) | 0.494 (2) | 0.0520 |
| H71 | 1.0000 | 0.3178 (13) | 1.0099 (10) | 0.4206 (9) | 0.0422 |
| H72 | 1.0000 | 0.4380 (13) | 0.9291 (10) | 0.3890 (9) | 0.0422 |
| H81 | 1.0000 | 0.3844 (13) | 1.1124 (10) | 0.2833 (9) | 0.0443 |
| H82 | 1.0000 | 0.3768 (13) | 0.9848 (11) | 0.2176 (9) | 0.0443 |
| H101 | 0.804 (12) | −0.1251 (15) | 0.7707 (12) | 0.0974 (10) | 0.0417 |
| H102 | 0.804 (12) | −0.2175 (15) | 0.6618 (13) | 0.1418 (10) | 0.0417 |
| H103 | 0.804 (12) | −0.0933 (15) | 0.6299 (12) | 0.0807 (10) | 0.0417 |
| H111 | 0.779 (13) | −0.0539 (16) | 0.4286 (13) | 0.3275 (11) | 0.0465 |
| H112 | 0.779 (13) | −0.1969 (15) | 0.5028 (13) | 0.2824 (11) | 0.0465 |
| H113 | 0.779 (13) | −0.0923 (16) | 0.4574 (13) | 0.2086 (11) | 0.0465 |
| H121 | 0.819 (13) | 0.3093 (16) | 0.7147 (13) | 0.5742 (11) | 0.0509 |
| H122 | 0.819 (13) | 0.3470 (16) | 0.8578 (13) | 0.5495 (11) | 0.0509 |
| H123 | 0.819 (13) | 0.4443 (16) | 0.7531 (13) | 0.5149 (11) | 0.0509 |
| H131 | 1.0000 | 0.1970 (14) | 1.1898 (11) | 0.1228 (9) | 0.0538 |
| H132 | 1.0000 | 0.0458 (14) | 1.0959 (11) | 0.0691 (9) | 0.0538 |
| H133 | 1.0000 | 0.2163 (14) | 1.0601 (11) | 0.0674 (9) | 0.0538 |
| H141 | 1.0000 | 0.1308 (13) | 1.1834 (11) | 0.2986 (9) | 0.0458 |
| H142 | 1.0000 | 0.0686 (13) | 1.0520 (11) | 0.3404 (9) | 0.0458 |
| H143 | 1.0000 | −0.0261 (13) | 1.1069 (11) | 0.2379 (9) | 0.0458 |
| H1011 | 0.5000 | 0.541 (3) | 0.511 (3) | 0.480 (2) | 0.0540 |
| H1012 | 0.5000 | 0.687 (2) | 0.516 (3) | 0.469 (2) | 0.0540 |
| H1071 | 1.0000 | 0.2939 (13) | 0.5041 (10) | 0.0664 (8) | 0.0391 |
| H1072 | 1.0000 | 0.1797 (12) | 0.5515 (10) | 0.1306 (8) | 0.0391 |

| | | | | | |
|---|---|---|---|---|---|
| H1081 | 1.0000 | 0.1796 (13) | 0.7415 (10) | 0.0552 (9) | 0.0416 |
| H1082 | 1.0000 | 0.1632 (12) | 0.6444 (10) | −0.0402 (9) | 0.0416 |
| H1101 | 0.698 (12) | 0.6951 (18) | 0.9330 (14) | 0.1868 (11) | 0.0443 |
| H1102 | 0.698 (12) | 0.8178 (16) | 0.9054 (14) | 0.2861 (12) | 0.0443 |
| H1103 | 0.698 (12) | 0.6811 (18) | 0.9800 (14) | 0.2985 (12) | 0.0443 |
| H1111 | 0.721 (13) | 0.7777 (17) | 0.7187 (14) | 0.5090 (11) | 0.0482 |
| H1112 | 0.721 (13) | 0.8832 (16) | 0.7642 (15) | 0.4329 (12) | 0.0482 |
| H1113 | 0.721 (13) | 0.7767 (18) | 0.8544 (13) | 0.4675 (12) | 0.0482 |
| H1121 | 0.713 (13) | 0.4055 (17) | 0.3786 (14) | 0.3384 (12) | 0.0476 |
| H1122 | 0.713 (13) | 0.3295 (18) | 0.3782 (14) | 0.2180 (11) | 0.0476 |
| H1123 | 0.713 (13) | 0.2535 (17) | 0.4367 (14) | 0.2999 (12) | 0.0476 |
| H1131 | 1.0000 | 0.2842 (14) | 0.8226 (11) | −0.1357 (10) | 0.0565 |
| H1132 | 1.0000 | 0.4349 (14) | 0.9085 (12) | −0.0738 (10) | 0.0565 |
| H1133 | 1.0000 | 0.2736 (14) | 0.9103 (12) | −0.0409 (10) | 0.0565 |
| H1141 | 1.0000 | 0.4112 (13) | 0.6204 (11) | −0.1010 (9) | 0.0465 |
| H1142 | 1.0000 | 0.5579 (13) | 0.7170 (11) | −0.0555 (9) | 0.0465 |
| H1143 | 1.0000 | 0.5097 (13) | 0.6050 (11) | 0.0126 (9) | 0.0465 |
| H104 | 0.196 (12) | −0.16176 (13) | 0.60207 (11) | 0.11744 (9) | 0.0403 |
| H105 | 0.196 (12) | −0.19888 (13) | 0.74482 (11) | 0.13239 (9) | 0.0403 |
| H106 | 0.196 (12) | −0.06778 (13) | 0.71829 (11) | 0.07187 (9) | 0.0403 |
| H114 | 0.221 (13) | −0.17694 (14) | 0.50054 (11) | 0.21764 (10) | 0.0460 |
| H115 | 0.221 (13) | −0.13240 (14) | 0.46978 (11) | 0.34002 (10) | 0.0460 |
| H116 | 0.221 (13) | −0.03181 (14) | 0.42521 (11) | 0.26210 (10) | 0.0460 |
| H124 | 0.181 (13) | 0.42375 (15) | 0.83518 (13) | 0.51700 (9) | 0.0486 |
| H125 | 0.181 (13) | 0.28821 (15) | 0.79623 (13) | 0.57698 (9) | 0.0486 |
| H126 | 0.181 (13) | 0.38215 (15) | 0.68923 (13) | 0.54284 (9) | 0.0486 |
| H1104 | 0.302 (12) | 0.76604 (13) | 0.94452 (11) | 0.33033 (10) | 0.0429 |
| H1105 | 0.302 (12) | 0.78306 (13) | 0.89804 (11) | 0.21637 (10) | 0.0429 |
| H1106 | 0.302 (12) | 0.63904 (13) | 0.97040 (11) | 0.22646 (10) | 0.0429 |
| H1114 | 0.279 (13) | 0.84494 (14) | 0.83901 (12) | 0.42716 (9) | 0.0467 |
| H1115 | 0.279 (13) | 0.84470 (14) | 0.69819 (12) | 0.47045 (9) | 0.0467 |
| H1116 | 0.279 (13) | 0.73759 (14) | 0.79345 (12) | 0.50605 (9) | 0.0467 |
| H1124 | 0.287 (13) | 0.25466 (15) | 0.41919 (11) | 0.22955 (10) | 0.0482 |
| H1125 | 0.287 (13) | 0.33044 (15) | 0.42093 (11) | 0.35380 (10) | 0.0482 |
| H1126 | 0.287 (13) | 0.41371 (15) | 0.36032 (11) | 0.27173 (10) | 0.0482 |

Table *S*2. *Anisotropic displacement parameters* (Å$^2$)

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0431 (5) | 0.0463 (5) | 0.0437 (5) | 0.0066 (4) | 0.0103 (4) | 0.0177 (4) |
| O2 | 0.0398 (4) | 0.0283 (4) | 0.0303 (4) | −0.0023 (3) | −0.0001 (3) | 0.0012 (3) |
| O101 | 0.0488 (5) | 0.0549 (6) | 0.0368 (5) | 0.0133 (5) | 0.0093 (4) | 0.0223 (4) |
| O102 | 0.0404 (4) | 0.0302 (4) | 0.0270 (4) | 0.0034 (3) | 0.0005 (3) | 0.0056 (3) |
| C1 | 0.0287 (5) | 0.0287 (5) | 0.0281 (5) | 0.0045 (4) | 0.0055 (4) | 0.0005 (4) |
| C2 | 0.0255 (5) | 0.0321 (5) | 0.0277 (5) | 0.0030 (4) | 0.0056 (4) | −0.0027 (4) |
| C3 | 0.0278 (5) | 0.0309 (5) | 0.0316 (5) | 0.0028 (4) | 0.0089 (4) | −0.0008 (4) |
| C4 | 0.0283 (5) | 0.0343 (6) | 0.0307 (5) | 0.0068 (4) | 0.0079 (4) | 0.0055 (4) |
| C5 | 0.0274 (5) | 0.0354 (6) | 0.0302 (5) | 0.0071 (4) | 0.0031 (4) | −0.0009 (4) |
| C6 | 0.0265 (5) | 0.0305 (5) | 0.0306 (5) | 0.0041 (4) | 0.0037 (4) | −0.0028 (4) |
| C7 | 0.0293 (5) | 0.0334 (6) | 0.0402 (6) | 0.0009 (5) | −0.0007 (5) | −0.0032 (5) |
| C8 | 0.0331 (6) | 0.0331 (6) | 0.0451 (7) | −0.0030 (5) | 0.0061 (5) | −0.0004 (5) |
| C9 | 0.0366 (6) | 0.0269 (5) | 0.0352 (6) | −0.0026 (4) | 0.0029 (5) | −0.0003 (4) |
| C10 | 0.0329 (6) | 0.0369 (6) | 0.0310 (6) | −0.0014 (5) | 0.0013 (5) | −0.0022 (5) |
| C11 | 0.0404 (6) | 0.0351 (6) | 0.0395 (6) | −0.0043 (5) | 0.0077 (5) | 0.0011 (5) |
| C12 | 0.0425 (7) | 0.0431 (7) | 0.0357 (6) | 0.0035 (5) | −0.0041 (5) | 0.0026 (5) |
| C13 | 0.0573 (8) | 0.0364 (7) | 0.0398 (7) | −0.0059 (6) | 0.0051 (6) | 0.0049 (5) |
| C14 | 0.0374 (6) | 0.0342 (6) | 0.0429 (7) | 0.0039 (5) | 0.0016 (5) | −0.0019 (5) |
| C101 | 0.0294 (5) | 0.0277 (5) | 0.0254 (5) | 0.0068 (4) | 0.0063 (4) | 0.0037 (4) |
| C102 | 0.0283 (5) | 0.0280 (5) | 0.0297 (5) | 0.0046 (4) | 0.0070 (4) | 0.0015 (4) |
| C103 | 0.0282 (5) | 0.0348 (6) | 0.0268 (5) | 0.0074 (4) | 0.0059 (4) | 0.0005 (4) |
| C104 | 0.0323 (5) | 0.0354 (6) | 0.0274 (5) | 0.0105 (4) | 0.0099 (4) | 0.0084 (4) |
| C105 | 0.0321 (5) | 0.0294 (5) | 0.0313 (5) | 0.0053 (4) | 0.0115 (4) | 0.0036 (4) |
| C106 | 0.0280 (5) | 0.0295 (5) | 0.0274 (5) | 0.0049 (4) | 0.0073 (4) | 0.0003 (4) |
| C107 | 0.0313 (6) | 0.0365 (6) | 0.0322 (6) | −0.0004 (5) | 0.0059 (5) | −0.0015 (5) |
| C108 | 0.0310 (6) | 0.0447 (7) | 0.0299 (6) | 0.0070 (5) | 0.0011 (4) | −0.0010 (5) |
| C109 | 0.0361 (6) | 0.0356 (6) | 0.0252 (5) | 0.0060 (5) | 0.0009 (4) | 0.0026 (4) |
| C110 | 0.0346 (6) | 0.0328 (6) | 0.0398 (6) | −0.0003 (5) | 0.0033 (5) | 0.0031 (5) |
| C111 | 0.0379 (6) | 0.0468 (7) | 0.0321 (6) | 0.0062 (5) | 0.0000 (5) | −0.0006 (5) |
| C112 | 0.0458 (7) | 0.0333 (6) | 0.0413 (7) | −0.0003 (5) | 0.0115 (5) | 0.0073 (5) |
| C113 | 0.0556 (8) | 0.0471 (7) | 0.0345 (6) | 0.0111 (6) | −0.0006 (6) | 0.0114 (6) |
| C114 | 0.0415 (7) | 0.0433 (7) | 0.0352 (6) | 0.0048 (5) | 0.0120 (5) | 0.0013 (5) |

Table *S*3. *Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1—C4 | 1.3950 (13) | C13—H132 | 1.002 (12) |
| O1—H11 | 0.814 (15) | C13—H133 | 1.001 (12) |
| O1—H12 | 0.818 (17) | C14—H141 | 0.978 (11) |
| O2—C1 | 1.3827 (12) | C14—H142 | 1.002 (11) |
| O2—C9 | 1.4566 (12) | C14—H143 | 0.972 (11) |
| O101—C104 | 1.3923 (13) | C101—C102 | 1.4012 (14) |
| O101—H1011 | 0.813 (15) | C101—C106 | 1.3926 (14) |
| O101—H1012 | 0.802 (17) | C102—C103 | 1.3963 (14) |
| O102—C101 | 1.3851 (12) | C102—C110 | 1.5019 (15) |
| O102—C109 | 1.4532 (12) | C103—C104 | 1.3979 (14) |
| C1—C2 | 1.4055 (14) | C103—C111 | 1.5074 (15) |
| C1—C6 | 1.3934 (14) | C104—C105 | 1.3884 (15) |
| C2—C3 | 1.3954 (14) | C105—C106 | 1.4023 (14) |
| C2—C10 | 1.5084 (14) | C105—C112 | 1.5086 (15) |
| C3—C4 | 1.3974 (15) | C106—C107 | 1.5112 (14) |
| C3—C11 | 1.5113 (15) | C107—C108 | 1.5212 (16) |
| C4—C5 | 1.3929 (15) | C107—H1071 | 1.004 (10) |
| C5—C6 | 1.4012 (15) | C107—H1072 | 0.990 (10) |
| C5—C12 | 1.5097 (15) | C108—C109 | 1.5233 (16) |
| C6—C7 | 1.5132 (14) | C108—H1081 | 0.975 (11) |
| C7—C8 | 1.5202 (16) | C108—H1082 | 0.998 (11) |
| C7—H71 | 0.995 (11) | C109—C113 | 1.5172 (16) |
| C7—H72 | 0.990 (11) | C109—C114 | 1.5200 (16) |
| C8—C9 | 1.5217 (16) | C110—H1101 | 0.986 (14) |
| C8—H81 | 0.990 (11) | C110—H1102 | 0.977 (14) |
| C8—H82 | 1.002 (11) | C110—H1103 | 0.994 (14) |
| C9—C13 | 1.5159 (16) | C110—H1104 | 1.000 |
| C9—C14 | 1.5207 (16) | C110—H1105 | 1.000 |
| C10—H101 | 0.959 (13) | C110—H1106 | 1.000 |
| C10—H102 | 0.985 (13) | C111—H1111 | 0.974 (14) |
| C10—H103 | 0.999 (12) | C111—H1112 | 0.999 (14) |
| C10—H104 | 1.000 | C111—H1113 | 0.988 (14) |
| C10—H105 | 1.000 | C111—H1114 | 1.000 |
| C10—H106 | 1.000 | C111—H1115 | 1.000 |
| C11—H111 | 0.990 (13) | C111—H1116 | 1.000 |
| C11—H112 | 0.998 (13) | C112—H1121 | 0.978 (14) |
| C11—H113 | 0.991 (13) | C112—H1122 | 0.982 (14) |
| C11—H114 | 1.000 | C112—H1123 | 0.988 (14) |
| C11—H115 | 1.000 | C112—H1124 | 1.000 |
| C11—H116 | 1.000 | C112—H1125 | 1.000 |
| C12—H121 | 0.970 (13) | C112—H1126 | 1.000 |
| C12—H122 | 1.004 (13) | C113—H1131 | 0.983 (12) |
| C12—H123 | 1.018 (13) | C113—H1132 | 0.988 (12) |
| C12—H124 | 1.000 | C113—H1133 | 0.987 (12) |
| C12—H125 | 1.000 | C114—H1141 | 0.980 (11) |
| C12—H126 | 1.000 | C114—H1142 | 0.964 (11) |
| C13—H131 | 0.994 (12) | C114—H1143 | 0.994 (11) |
| C4—O1—H11 | 121 (2) | C4—C5—C6 | 118.86 (9) |
| C4—O1—H12 | 119 (2) | C4—C5—C12 | 121.11 (10) |
| H11—O1—H12 | 120 (3) | C6—C5—C12 | 120.03 (10) |
| C1—O2—C9 | 117.42 (8) | C5—C6—C1 | 119.19 (9) |
| C104—O101—H1011 | 121 (2) | C5—C6—C7 | 120.66 (9) |
| C104—O101—H1012 | 122 (2) | C1—C6—C7 | 120.15 (9) |
| H1011—O101—H1012 | 116 (3) | C6—C7—C8 | 111.53 (9) |
| C101—O102—C109 | 116.67 (8) | C6—C7—H71 | 109.8 (7) |
| O2—C1—C2 | 115.63 (9) | C8—C7—H71 | 109.6 (6) |
| O2—C1—C6 | 122.69 (9) | C6—C7—H72 | 108.4 (7) |
| C2—C1—C6 | 121.67 (9) | C8—C7—H72 | 110.0 (7) |
| C1—C2—C3 | 118.91 (9) | H71—C7—H72 | 107.4 (9) |
| C1—C2—C10 | 119.96 (9) | C7—C8—C9 | 111.31 (9) |
| C3—C2—C10 | 121.13 (9) | C7—C8—H81 | 110.8 (7) |
| C2—C3—C4 | 119.09 (9) | C9—C8—H81 | 109.4 (7) |
| C2—C3—C11 | 120.68 (10) | C7—C8—H82 | 110.9 (7) |
| C4—C3—C11 | 120.23 (10) | C9—C8—H82 | 107.6 (7) |
| C3—C4—O1 | 118.57 (9) | H81—C8—H82 | 106.7 (9) |
| C3—C4—C5 | 122.08 (10) | C8—C9—O2 | 107.95 (9) |
| O1—C4—C5 | 119.35 (9) | C8—C9—C13 | 111.68 (10) |

| | |
|---|---|
| O2—C9—C13 | 104.50 (9) |
| C8—C9—C14 | 112.69 (9) |
| O2—C9—C14 | 109.15 (9) |
| C13—C9—C14 | 110.49 (10) |
| C2—C10—H101 | 110.5 (8) |
| C2—C10—H102 | 110.1 (8) |
| H101—C10—H102 | 107.0 (10) |
| C2—C10—H103 | 112.1 (8) |
| H101—C10—H103 | 109.5 (10) |
| H102—C10—H103 | 107.5 (10) |
| C2—C10—H104 | 109.5 |
| C2—C10—H105 | 109.5 |
| H104—C10—H105 | 109.5 |
| C2—C10—H106 | 109.5 |
| H104—C10—H106 | 109.5 |
| H105—C10—H106 | 109.5 |
| C3—C11—H111 | 111.3 (8) |
| C3—C11—H112 | 111.2 (8) |
| H111—C11—H112 | 108.4 (11) |
| C3—C11—H113 | 110.5 (8) |
| H111—C11—H113 | 106.0 (11) |
| H112—C11—H113 | 109.4 (11) |
| C3—C11—H114 | 109.5 |
| H111—C11—H114 | 139.3 |
| H112—C11—H114 | 54.9 |
| H113—C11—H114 | 58.7 |
| C3—C11—H115 | 109.5 |
| H114—C11—H115 | 109.5 |
| C3—C11—H116 | 109.5 |
| H114—C11—H116 | 109.5 |
| H115—C11—H116 | 109.5 |
| C5—C12—H121 | 111.2 (8) |
| C5—C12—H122 | 110.0 (8) |
| H121—C12—H122 | 107.8 (11) |
| C5—C12—H123 | 110.4 (8) |
| H121—C12—H123 | 109.3 (11) |
| H122—C12—H123 | 108.1 (10) |
| C5—C12—H124 | 109.5 |
| H121—C12—H124 | 139.4 |
| H122—C12—H124 | 56.8 |
| H123—C12—H124 | 54.7 |
| C5—C12—H125 | 109.5 |
| H121—C12—H125 | 55.9 |
| H122—C12—H125 | 55.5 |
| H123—C12—H125 | 140.1 |
| H124—C12—H125 | 109.5 |
| C5—C12—H126 | 109.5 |
| H121—C12—H126 | 56.0 |
| H122—C12—H126 | 140.5 |
| H123—C12—H126 | 57.4 |
| H124—C12—H126 | 109.5 |
| H125—C12—H126 | 109.5 |
| C9—C13—H131 | 110.2 (7) |
| C9—C13—H132 | 110.8 (7) |
| H131—C13—H132 | 107.4 (10) |
| C9—C13—H133 | 109.9 (7) |
| H131—C13—H133 | 108.0 (10) |
| H132—C13—H133 | 110.5 (10) |
| C9—C14—H141 | 108.1 (7) |
| C9—C14—H142 | 112.2 (7) |
| H141—C14—H142 | 109.4 (9) |
| C9—C14—H143 | 110.4 (7) |
| H141—C14—H143 | 108.5 (9) |
| H142—C14—H143 | 108.2 (9) |
| O102—C101—C102 | 115.28 (9) |
| O102—C101—C106 | 122.66 (9) |
| C102—C101—C106 | 122.06 (9) |
| C101—C102—C103 | 118.98 (9) |
| C101—C102—C110 | 119.91 (9) |
| C103—C102—C110 | 121.11 (9) |
| C102—C103—C104 | 118.67 (9) |
| C102—C103—C111 | 121.23 (10) |
| C104—C103—C111 | 120.10 (10) |
| C103—C104—O101 | 118.70 (9) |
| C103—C104—C105 | 122.47 (9) |
| O101—C104—C105 | 118.83 (10) |
| C104—C105—C106 | 118.91 (9) |
| C104—C105—C112 | 120.74 (10) |
| C106—C105—C112 | 120.35 (10) |
| C105—C106—C101 | 118.85 (9) |
| C105—C106—C107 | 120.80 (9) |
| C101—C106—C107 | 120.35 (9) |
| C106—C107—C108 | 111.43 (9) |
| C106—C107—H1071 | 108.5 (6) |
| C108—C107—H1071 | 110.4 (6) |
| C106—C107—H1072 | 109.3 (6) |
| C108—C107—H1072 | 109.4 (6) |
| H1071—C107—H1072 | 107.7 (8) |
| C107—C108—C109 | 111.27 (9) |
| C107—C108—H1081 | 110.2 (7) |
| C109—C108—H1081 | 107.6 (7) |
| C107—C108—H1082 | 110.8 (6) |
| C109—C108—H1082 | 109.1 (6) |
| H1081—C108—H1082 | 107.8 (9) |
| C108—C109—O102 | 107.72 (8) |
| C108—C109—C113 | 111.45 (10) |
| O102—C109—C113 | 104.83 (9) |
| C108—C109—C114 | 113.33 (10) |
| O102—C109—C114 | 108.67 (9) |
| C113—C109—C114 | 110.42 (10) |
| C102—C110—H1101 | 111.3 (9) |
| C102—C110—H1102 | 110.2 (9) |
| H1101—C110—H1102 | 106.7 (12) |
| C102—C110—H1103 | 112.6 (9) |
| H1101—C110—H1103 | 107.5 (11) |
| H1102—C110—H1103 | 108.3 (11) |
| C102—C110—H1104 | 109.5 |
| C102—C110—H1105 | 109.5 |
| H1104—C110—H1105 | 109.5 |
| C102—C110—H1106 | 109.5 |
| H1104—C110—H1106 | 109.5 |
| H1105—C110—H1106 | 109.5 |
| C103—C111—H1111 | 112.2 (9) |
| C103—C111—H1112 | 112.5 (9) |
| H1111—C111—H1112 | 107.5 (11) |
| C103—C111—H1113 | 111.6 (9) |
| H1111—C111—H1113 | 106.6 (11) |
| H1112—C111—H1113 | 105.9 (11) |
| C103—C111—H1114 | 109.5 |
| C103—C111—H1115 | 109.5 |
| H1114—C111—H1115 | 109.5 |
| C103—C111—H1116 | 109.5 |
| H1114—C111—H1116 | 109.5 |
| H1115—C111—H1116 | 109.5 |
| C105—C112—H1121 | 112.5 (9) |
| C105—C112—H1122 | 112.3 (9) |
| H1121—C112—H1122 | 108.3 (12) |
| C105—C112—H1123 | 111.4 (9) |
| H1121—C112—H1123 | 105.7 (11) |
| H1122—C112—H1123 | 106.3 (11) |
| C105—C112—H1124 | 109.5 |
| C105—C112—H1125 | 109.5 |
| H1124—C112—H1125 | 109.5 |
| C105—C112—H1126 | 109.5 |
| H1124—C112—H1126 | 109.5 |
| H1125—C112—H1126 | 109.5 |
| C109—C113—H1131 | 109.3 (7) |
| C109—C113—H1132 | 110.6 (7) |

| | | | |
|---|---|---|---|
| H1131—C113—H1132 | 107.8 (10) | C109—C114—H1142 | 110.1 (7) |
| C109—C113—H1133 | 110.8 (7) | H1141—C114—H1142 | 108.1 (9) |
| H1131—C113—H1133 | 107.5 (10) | C109—C114—H1143 | 111.2 (7) |
| H1132—C113—H1133 | 110.8 (10) | H1141—C114—H1143 | 107.2 (9) |
| C109—C114—H1141 | 110.4 (7) | H1142—C114—H1143 | 109.7 (9) |

| | | | |
|---|---|---|---|
| O1—C4—C3—C2 | 177.2 (1) | C5—C4—C3—C11 | 177.8 (1) |
| O1—C4—C3—C11 | −2.3 (2) | C5—C6—C7—C8 | 168.3 (1) |
| O1—C4—C5—C6 | 179.3 (1) | C6—C1—O2—C9 | −15.8 (2) |
| O1—C4—C5—C12 | −0.5 (2) | C6—C1—C2—C10 | −178.6 (1) |
| O2—C1—C2—C3 | 179.8 (1) | C6—C7—C8—C9 | 43.5 (1) |
| O2—C1—C2—C10 | 0.3 (2) | C7—C6—C5—C12 | 5.0 (2) |
| O2—C1—C6—C5 | 176.8 (1) | C7—C8—C9—C13 | −176.2 (1) |
| O2—C1—C6—C7 | −4.1 (2) | C7—C8—C9—C14 | 58.8 (1) |
| O2—C9—C8—C7 | −61.8 (1) | C10—C2—C3—C11 | 1.6 (2) |
| O101—C104—C103—C102 | 178.4 (1) | C101—O102—C109—C108 | 50.3 (1) |
| O101—C104—C103—C111 | −1.2 (2) | C101—O102—C109—C113 | 169.1 (1) |
| O101—C104—C105—C106 | 179.7 (1) | C101—O102—C109—C114 | −72.9 (1) |
| O101—C104—C105—C112 | −0.6 (2) | C101—C102—C103—C104 | 1.5 (2) |
| O102—C101—C102—C103 | −179.8 (1) | C101—C102—C103—C111 | −178.9 (1) |
| O102—C101—C102—C110 | −0.3 (2) | C101—C106—C105—C104 | 2.2 (2) |
| O102—C101—C106—C105 | 177.9 (1) | C101—C106—C105—C112 | −177.5 (1) |
| O102—C101—C106—C107 | −2.0 (2) | C101—C106—C107—C108 | −11.0 (2) |
| O102—C109—C108—C107 | −62.5 (1) | C102—C101—O102—C109 | 161.4 (1) |
| C1—O2—C9—C8 | 47.8 (1) | C102—C101—C106—C105 | −2.6 (2) |
| C1—O2—C9—C13 | 166.8 (1) | C102—C101—C106—C107 | 177.5 (1) |
| C1—O2—C9—C14 | −75.0 (1) | C102—C103—C104—C105 | −1.9 (2) |
| C1—C2—C3—C4 | 2.6 (2) | C103—C102—C101—C106 | 0.7 (2) |
| C1—C2—C3—C11 | −177.9 (1) | C103—C104—C105—C106 | 0.0 (2) |
| C1—C6—C5—C4 | 4.3 (2) | C103—C104—C105—C112 | 179.7 (1) |
| C1—C6—C5—C12 | −175.8 (1) | C104—C103—C102—C110 | −178.0 (1) |
| C1—C6—C7—C8 | −10.9 (2) | C104—C105—C106—C107 | −177.9 (1) |
| C2—C1—O2—C9 | 165.3 (1) | C105—C104—C103—C111 | 178.5 (1) |
| C2—C1—C6—C5 | −4.4 (2) | C105—C106—C107—C108 | 169.1 (1) |
| C2—C1—C6—C7 | 174.7 (1) | C106—C101—O102—C109 | −19.2 (2) |
| C2—C3—C4—C5 | −2.7 (2) | C106—C101—C102—C110 | −179.8 (1) |
| C3—C2—C1—C6 | 0.9 (2) | C106—C107—C108—C109 | 42.8 (1) |
| C3—C4—C5—C6 | −0.8 (2) | C107—C106—C105—C112 | 2.4 (2) |
| C3—C4—C5—C12 | 179.3 (1) | C107—C108—C109—C113 | −177.0 (1) |
| C4—C3—C2—C10 | −177.9 (1) | C107—C108—C109—C114 | 57.7 (1) |
| C4—C5—C6—C7 | −174.8 (1) | C110—C102—C103—C111 | 1.6 (2) |

Table *S*4. *Contact distances (Å)*

| | | | |
|---|---|---|---|
| O1···O101$^{i}$ | 2.683 (1) | O101···C112$^{i}$ | 3.531 (2) |
| O1···O1$^{ii}$ | 2.713 (2) | O101···C104$^{i}$ | 3.539 (2) |
| O1···C11$^{ii}$ | 3.476 (2) | O101···C4$^{i}$ | 3.573 (1) |
| O101···O101$^{i}$ | 2.634 (2) | C10···C102$^{iii}$ | 3.594 (2) |
| O101···C12$^{i}$ | 3.361 (2) | | |

Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $-x, 1-y, 1-z$; (iii) $x-1, y, z$.

Table of Least-Squares Planes

---

---------- Plane number 1 ----------

| Atoms Defining Plane | | | Distance | esd |
|---|---|---|---|---|
| C1 | ( | 1) | -0.0151 | 0.0011 |
| C2 | ( | 1) | -0.0083 | 0.0012 |
| C3 | ( | 1) | 0.0226 | 0.0012 |
| C4 | ( | 1) | -0.0097 | 0.0012 |
| C5 | ( | 1) | -0.0165 | 0.0012 |
| C6 | ( | 1) | 0.0274 | 0.0012 |

| Additional Atoms | | | Distance |
|---|---|---|---|
| O1 | ( | 1) | -0.0391 |
| O2 | ( | 1) | -0.0421 |
| C7 | ( | 1) | 0.1395 |
| C8 | ( | 1) | 0.4572 |
| C9 | ( | 1) | -0.3783 |
| C10 | ( | 1) | -0.0224 |
| C11 | ( | 1) | 0.0785 |
| C12 | ( | 1) | -0.0664 |
| C13 | ( | 1) | -0.0027 |
| C14 | ( | 1) | -1.8729 |

Mean deviation from plane is 0.0166 angstroms
Chi-squared: 1400.1

---------- Plane number 2 ----------

| Atoms Defining Plane | | | Distance | esd |
|---|---|---|---|---|
| O2 | ( | 1) | 0.0793 | 0.0009 |
| C1 | ( | 1) | 0.0387 | 0.0011 |
| C6 | ( | 1) | -0.0770 | 0.0012 |
| C7 | ( | 1) | -0.0709 | 0.0013 |
| C8 | ( | 1) | 0.3331 | 0.0014 |
| C9 | ( | 1) | -0.3518 | 0.0013 |

| Additional Atoms | | | Distance |
|---|---|---|---|
| O1 | ( | 1) | -0.1600 |
| C2 | ( | 1) | 0.1470 |
| C3 | ( | 1) | 0.1180 |
| C4 | ( | 1) | -0.0727 |
| C5 | ( | 1) | -0.1802 |
| C10 | ( | 1) | 0.3033 |
| C11 | ( | 1) | 0.2785 |
| C12 | ( | 1) | -0.4007 |
| C13 | ( | 1) | 0.1156 |
| C14 | ( | 1) | -1.8687 |

Mean deviation from plane is 0.1585 angstroms
Chi-squared: 147967.0

Dihedral angles between least-squares planes

| plane | plane | angle |
|---|---|---|
| 2 | 1 | 6.57 |

Table of Least-Squares Planes (continued)

---------- Plane number 3 ----------

| Atoms Defining Plane | | | Distance | esd |
|---|---|---|---|---|
| O2 | ( | 1) | -0.0054 | 0.0009 |
| C1 | ( | 1) | 0.0175 | 0.0011 |
| C6 | ( | 1) | -0.0173 | 0.0012 |
| C7 | ( | 1) | 0.0106 | 0.0013 |

| Additional Atoms | | | Distance |
|---|---|---|---|
| O1 | ( | 1) | -0.0017 |
| C2 | ( | 1) | 0.1037 |
| C3 | ( | 1) | 0.1356 |
| C4 | ( | 1) | 0.0256 |
| C5 | ( | 1) | -0.0599 |
| C8 | ( | 1) | 0.3420 |
| C9 | ( | 1) | -0.4191 |
| C10 | ( | 1) | 0.1718 |
| C11 | ( | 1) | 0.2753 |
| C12 | ( | 1) | -0.1928 |
| C13 | ( | 1) | -0.0255 |
| C14 | ( | 1) | -1.9270 |

Mean deviation from plane is 0.0127 angstroms
Chi-squared: 565.0

Dihedral angles between least-squares planes

| plane | plane | angle |
|---|---|---|
| 3 | 1 | 3.68 |
| 3 | 2 | 3.47 |

---------- Plane number 4 ----------

| Atoms Defining Plane | | | Distance | esd |
|---|---|---|---|---|
| C101 | ( | 1) | 0.0098 | 0.0011 |
| C102 | ( | 1) | 0.0036 | 0.0011 |
| C103 | ( | 1) | -0.0131 | 0.0012 |
| C104 | ( | 1) | 0.0085 | 0.0012 |
| C105 | ( | 1) | 0.0075 | 0.0012 |
| C106 | ( | 1) | -0.0155 | 0.0011 |

| Additional Atoms | | | Distance |
|---|---|---|---|
| O101 | ( | 1) | 0.0252 |
| O102 | ( | 1) | 0.0316 |
| C107 | ( | 1) | -0.0653 |
| C108 | ( | 1) | -0.3503 |
| C109 | ( | 1) | 0.4539 |
| C110 | ( | 1) | 0.0262 |
| C111 | ( | 1) | -0.0457 |
| C112 | ( | 1) | 0.0376 |
| C113 | ( | 1) | 0.1178 |
| C114 | ( | 1) | 1.9546 |

Mean deviation from plane is 0.0097 angstroms
Chi-squared: 499.0

Dihedral angles between least-squares planes

| plane | plane | angle |
|---|---|---|
| 4 | 1 | 2.94 |
| 4 | 2 | 6.19 |
| 4 | 3 | 2.72 |

Table of Least-Squares Planes (continued)

---------- Plane number 5 ----------

| Atoms Defining Plane | | | Distance | esd |
|---|---|---|---|---|
| O102 | ( | 1) | -0.0974 | 0.0009 |
| C101 | ( | 1) | -0.0184 | 0.0011 |
| C106 | ( | 1) | 0.0788 | 0.0011 |
| C107 | ( | 1) | 0.0571 | 0.0013 |
| C108 | ( | 1) | -0.3432 | 0.0014 |
| C109 | ( | 1) | 0.3472 | 0.0012 |

| Additional Atoms | | | Distance |
|---|---|---|---|
| O101 | ( | 1) | 0.2818 |
| C102 | ( | 1) | -0.0543 |
| C103 | ( | 1) | 0.0252 |
| C104 | ( | 1) | 0.1694 |
| C105 | ( | 1) | 0.1981 |
| C110 | ( | 1) | -0.1654 |
| C111 | ( | 1) | -0.0339 |
| C112 | ( | 1) | 0.3618 |
| C113 | ( | 1) | -0.1053 |
| C114 | ( | 1) | 1.8639 |

Mean deviation from plane is 0.1570 angstroms
Chi-squared: 168641.9

Dihedral angles between least-squares planes

| plane | plane | angle |
|---|---|---|
| 5 | 1 | 8.21 |
| 5 | 2 | 9.47 |
| 5 | 3 | 6.89 |
| 5 | 4 | 5.33 |

---------- Plane number 6 ----------

| Atoms Defining Plane | | | Distance | esd |
|---|---|---|---|---|
| O102 | ( | 1) | 0.0025 | 0.0009 |
| C101 | ( | 1) | -0.0083 | 0.0011 |
| C106 | ( | 1) | 0.0084 | 0.0011 |
| C107 | ( | 1) | -0.0054 | 0.0013 |

| Additional Atoms | | | Distance |
|---|---|---|---|
| O101 | ( | 1) | 0.0317 |
| C102 | ( | 1) | -0.0487 |
| C103 | ( | 1) | -0.0571 |
| C104 | ( | 1) | 0.0068 |
| C105 | ( | 1) | 0.0397 |
| C108 | ( | 1) | -0.3066 |
| C109 | ( | 1) | 0.4573 |
| C110 | ( | 1) | -0.0708 |
| C111 | ( | 1) | -0.1252 |
| C112 | ( | 1) | 0.1149 |
| C113 | ( | 1) | 0.1038 |
| C114 | ( | 1) | 1.9635 |

Mean deviation from plane is 0.0062 angstroms
Chi-squared: 140.5

Dihedral angles between least-squares planes

| plane | plane | angle |
|---|---|---|
| 6 | 1 | 4.68 |
| 6 | 2 | 5.93 |
| 6 | 3 | 2.90 |
| 6 | 4 | 1.82 |
| 6 | 5 | 4.03 |

**Figure Captions for $C_{14}H_{20}O_2$**

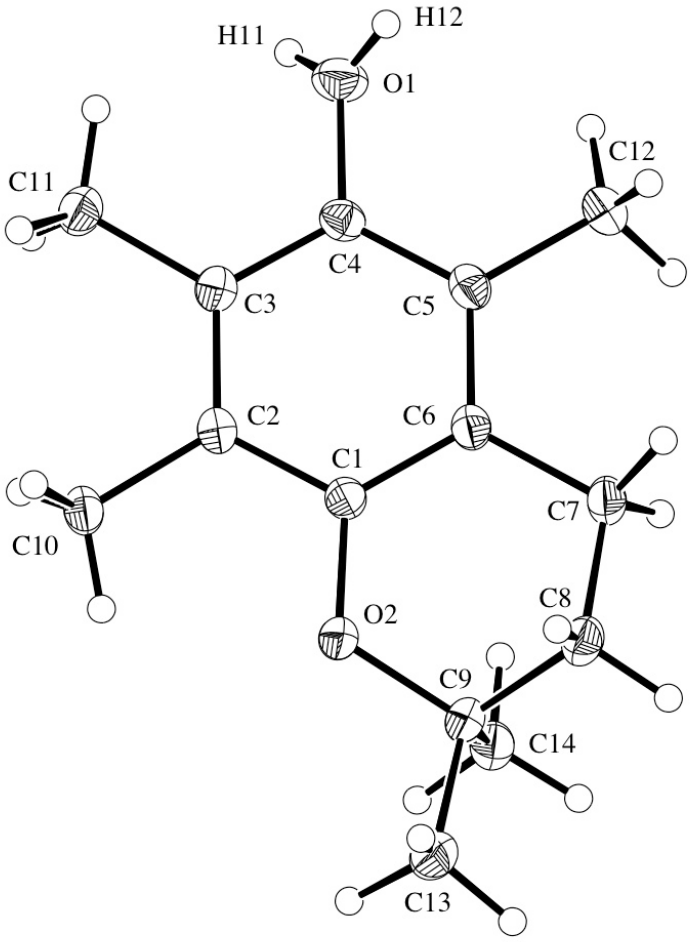


**Figure 1**. Structure of molecule one of $C_{14}H_{20}O_2$ with labelling of selected atoms. Anisotropic displacement ellipsoids show 30% probability levels. Hydrogen atoms are drawn as circles with small radii; only the major sites are shown for methyl groups, but both half-occupancy sites are displayed for alcohol hydrogens.

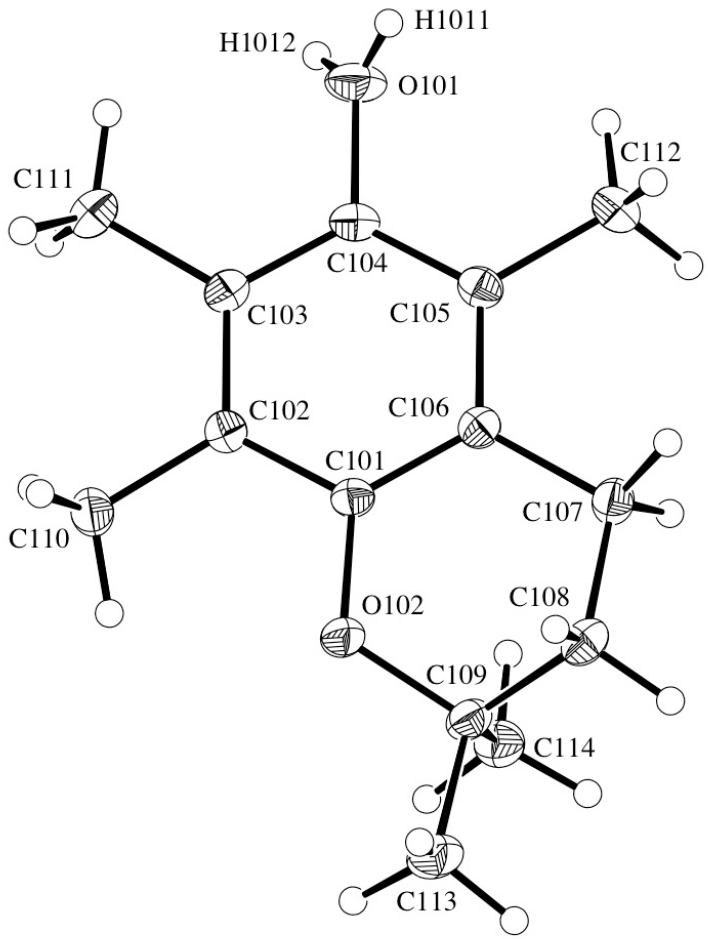


**Figure 2**. Structure of molecule two of $C_{14}H_{20}O_2$ with labelling of selected atoms. Anisotropic displacement ellipsoids show 30% probability levels. Hydrogen atoms are drawn as circles with small radii; only the major sites are shown for methyl groups, but both half-occupancy sites are displayed for alcohol hydrogens.

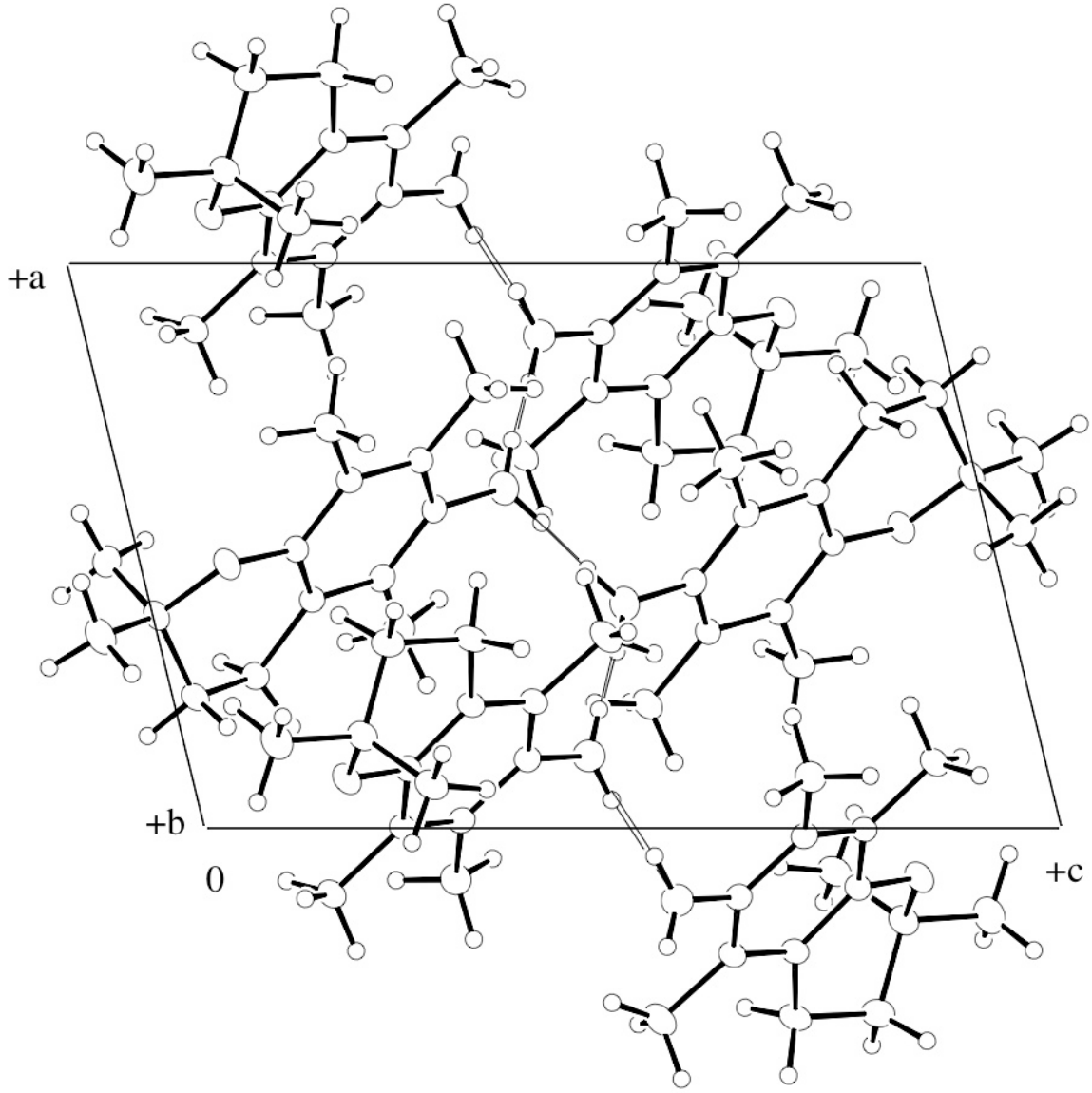


**Figure 3**. Unit cell packing diagram of $C_{14}H_{20}O_2$ projected down the *a* axis. Hydrogen atoms are drawn as circles with small radii; only the major sites are shown for methyl groups, but both half-occupancy sites are displayed for alcohol hydrogens.

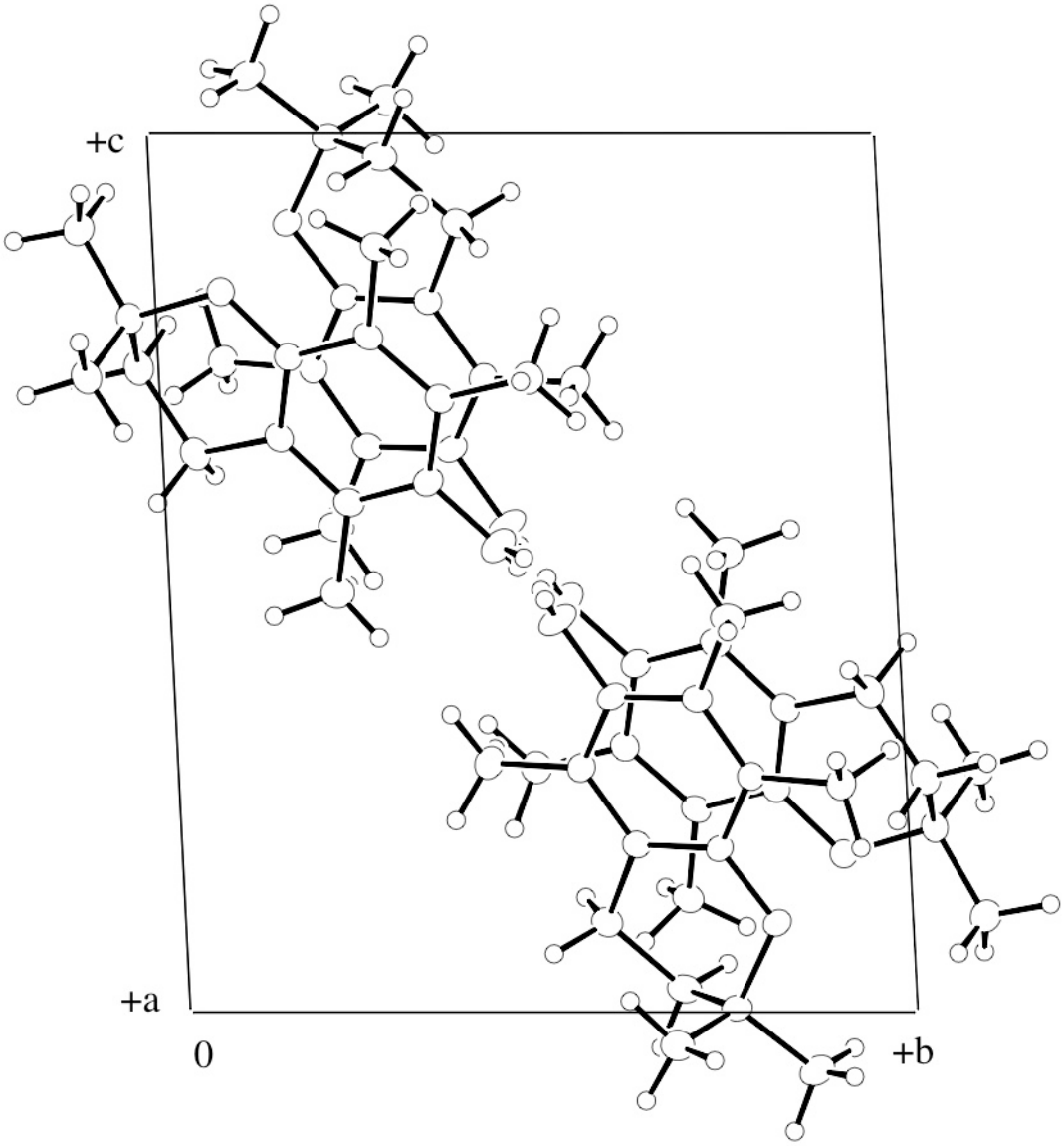


**Figure 4**. Unit cell packing diagram of $C_{14}H_{20}O_2$ projected down the *b* axis, showing hydrogen-bonding. Hydrogen atoms are drawn as circles with small radii; only the major sites are shown for methyl groups, but both half-occupancy sites are displayed for alcohol hydrogens.