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# Trichodermin (12,13-epoxytrichethec-9-en-4$\beta$-yl 4-fluorobenzoate)

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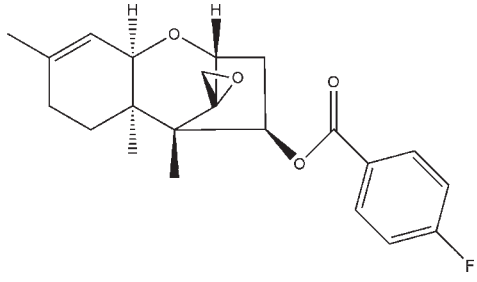
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.034; $wR$ factor = 0.086; data-to-parameter ratio = 8.8.

In the title trichodermin compound (systematic name: 12,13-epoxytrichothec-9-en-4$\beta$-yl 4-fluorobenzoate), $C_{22}H_{25}FO_4$, the five-membered ring displays an envelope conformation, whereas the two six-membered rings show the different conformations, *viz*. chair and half-chair. As for the seven-membered ring, the dihedral angle between the mean planes formed by the four C atoms of the envelope unit and the three C and one O atoms of the six-membered chair is 68.67 (2)°; these two mean planes are nearly perpendicular to the epoxy ring with angles of 87.97 (2)and 88.14 (2)°, respectively.

## Related literature

The endophytic fungi Trichoderma taxi *sp*. nov. can produce a compound with fungicidal activity, Trichodermin (Zhang *et al.*, 2007), which is a member of the 4$\beta$-acetoxy-12,13-epoxy-trichothecene family (Nielsen *et al.*, 2005). For a related Trichodermin structure, see: Chen *et al.* (2008). For the structures of Trichodermin derivatives, see: Cheng *et al.* (2009); Zhao *et al.* (2010). For the extinction correction, see: Larson (1970).




## Experimental

### Crystal data

$C_{22}H_{25}FO_4$
$M_r$ = 372.42
Orthorhombic, $P2_12_12_1$
$a$ = 8.1643 (4) Å
$b$ = 9.9979 (4) Å
$c$ = 23.6503 (9) Å
$V$ = 1930.48 (14) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 296 K
0.49 × 0.38 × 0.28 mm

### Data collection

Rigaku R-AXIS RAPID/ZJUG diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min}$ = 0.956, $T_{max}$ = 0.974
16756 measured reflections
2183 independent reflections
1760 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.030

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.086
$S$ = 1.00
2183 reflections
248 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.16 e Å$^{-3}$

Data collection: *PROCESS-AUTO* (Rigaku, 2006); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2275).

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# supporting information

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## Trichodermin (12,13-epoxytrichethec-9-en-4*β*-yl 4-fluorobenzoate)

**Xu-hui Xu, Zong-cheng Wang, Jing-li Cheng, Yong Zhou and Jin-hao Zhao**

### S1. Comment

The endophytic fungi Trichoderma taxi *sp*. nov. from Taxus mairei S.Y.Hu can produce a compound with fungicidal activity-Trichodermin (Zhang *et al.*, 2007), which is a member of the 4*β*-acetoxy-12,13-epoxytrichothecene family (Nielsen *et al.*, 2005). Bioassays showed Trichodermin strongly inhibited Rhizoctonia solani and Botrytis cinere. Furthermore, addition of bromine at the C9=C10 double bond led to the absolutely loss in antifungal activity (Zhao *et al.*, 2010). 4*β* Conformation and its acetate are key moieties for its bioactivities (Zhang *et al.*, 2007; Cheng *et al.*, 2009). Therefore, in order to develop novel, safe and potential microbial pesticides, the 4*β* substituted phenyl esters such as the title compound were designed and synthesized. Its molecular structure is shown in Fig. 1. In the molecule, the five membered ring displays an envelope conformation with C11 atom at the flap position 0.695 (3) Å out of the mean plane formed by the other four atoms. The two six-membered rings display different conformations. The O1-containing ring displays a chair conformation. Whereas the C6-containing six-membered ring displays a half-chair conformation. As for the seven-membered ring, the dihedral angle between the mean planes formed by C1,C2,C3,C10 and C3,O1,C9,C10 is 68.67 (2) °, which are nearly perpendicular to the epoxy ring with angles of 87.97 (2) and 88.14 (2) °, respectively.

### S2. Experimental

In a flask, 4*β*-hydroxy-12,13-epoxytrichethec-9-ene 2.50 g (10 mmol) were introduced with 40 ml of absolute methylene chloride, and then 1.34 g (11 mmol) of triethylamine was added. After the mixture was dissolved, a solution of 1.73 g (11 mmol) of 4-fluorobenzoyl chloride in 10 ml of absolute methylene chloride was added dropwise in 20 min at 273–278 K. After stirring for 2 h, the reaction solution was washed with 1% HCl solution, sat. $NaHCO_3$, and sat. NaCl solution respectively. After dried with sodium sulfate, the solution was concentrated. The residue was crystallized and purified with 95% ethanol to colourless blocks. The 1H NMR,ESI-MS data testified the title compound's structure. 1*H*-NMR (500 MHz, $CDCl_3$, ppm): 8.09–7.09 (4*H*, m, H-4), 5.80–5.77 (1*H*, m, H-4), 5.44–5.43 (1*H*, m, H-10), 3.89 (1*H*, d, J=5.5 Hz, H-2), 3.68 (1*H*, d, J=5.5 Hz, H-11), 3.17 (1*H*, d, J=4.0 Hz, H-13), 2.87 (1*H*, d, J=4.0 Hz, H-13), 2.67–2.63 (1*H*, m, H-3), 2.15–2.11 (1*H*, m, H-3), 2.03 (2*H*, s, H-8), 2.01–1.95 (1*H*, m, H-7), 1.73 (3*H*, s, H-16), 1.46–1.43 (1*H*, m, H-7), 0.98 (3*H*, s, H-14), 0.792 (3*H*, s, H-15); ESI-MS: 372 (*M*+H)+, (100%).

### S3. Refinement

In the absence of significant anomalous scattering effects, Friedel pairs were merged; the absolute configuration was not determined. The H atoms were geometrically placed (C–H = 0.93–0.98 Å) and refined as riding with $U_{iso}$(H) = 1.2$U_{eq}$(C).

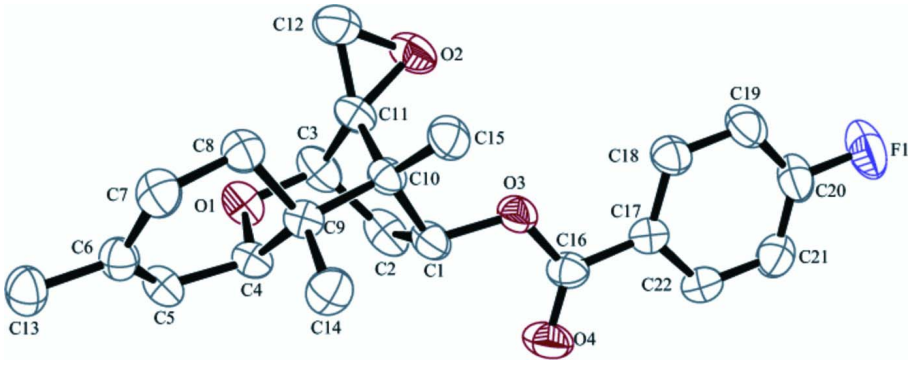


**Figure 1**

The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 40% probability level.

## 12,13-epoxytrichothec-9-en-4$\beta$-yl 4-fluorobenzoate

*Crystal data*

$C_{22}H_{25}FO_4$
$M_r = 372.42$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 8.1643$ (4) Å
$b = 9.9979$ (4) Å
$c = 23.6503$ (9) Å
$V = 1930.48$ (14) Å$^3$
$Z = 4$
$F(000) = 792$
$D_x = 1.281$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 13225 reflections
$\theta = 3.0$–$27.4°$
$\mu = 0.09$ mm$^{-1}$
$T = 296$ K
Chunk, colorless
0.49 × 0.38 × 0.28 mm

*Data collection*

Rigaku R-AXIS RAPID/ZJUG diffractometer
Radiation source: rolling anode
Graphite monochromator
Detector resolution: 10.00 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.956$, $T_{max} = 0.974$
16756 measured reflections
2183 independent reflections
1760 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 26.0°$, $\theta_{min} = 3.0°$
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 12$
$l = -29 \rightarrow 29$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.086$
$S = 1.00$
2183 reflections
248 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0289P)^2 + 0.6417P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.16$ e Å$^{-3}$
$\Delta\rho_{min} = -0.16$ e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0158 (10)

*Special details*

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

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on $F^2$, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^2$ are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O3 | 0.5928 (2) | 0.17729 (19) | 0.38572 (8) | 0.0590 (5) |
| O1 | 0.3925 (2) | 0.58802 (17) | 0.41271 (7) | 0.0552 (5) |
| C9 | 0.3220 (3) | 0.4541 (3) | 0.32881 (10) | 0.0448 (6) |
| C10 | 0.3644 (3) | 0.3262 (2) | 0.36490 (10) | 0.0452 (6) |
| C4 | 0.4116 (3) | 0.5769 (2) | 0.35259 (9) | 0.0467 (6) |
| H4 | 0.5286 | 0.5683 | 0.3440 | 0.056* |
| O4 | 0.8524 (3) | 0.2138 (2) | 0.35947 (10) | 0.0769 (7) |
| C17 | 0.7821 (3) | 0.0043 (3) | 0.40042 (10) | 0.0481 (6) |
| C6 | 0.2105 (4) | 0.7223 (3) | 0.30230 (11) | 0.0516 (7) |
| C8 | 0.1381 (3) | 0.4869 (3) | 0.33098 (11) | 0.0529 (7) |
| H8A | 0.0763 | 0.4100 | 0.3179 | 0.063* |
| H8B | 0.1066 | 0.5044 | 0.3698 | 0.063* |
| C5 | 0.3514 (4) | 0.7072 (3) | 0.32885 (11) | 0.0514 (7) |
| H5 | 0.4177 | 0.7821 | 0.3331 | 0.062* |
| C18 | 0.6592 (4) | −0.0777 (3) | 0.42095 (12) | 0.0576 (7) |
| H18 | 0.5513 | −0.0480 | 0.4210 | 0.069* |
| O2 | 0.2982 (3) | 0.2494 (2) | 0.46425 (8) | 0.0710 (6) |
| C7 | 0.0950 (4) | 0.6076 (3) | 0.29473 (12) | 0.0607 (7) |
| H7A | 0.0952 | 0.5810 | 0.2553 | 0.073* |
| H7B | −0.0149 | 0.6369 | 0.3041 | 0.073* |
| C16 | 0.7495 (3) | 0.1413 (3) | 0.37958 (11) | 0.0521 (7) |
| C22 | 0.9427 (4) | −0.0412 (3) | 0.40068 (11) | 0.0565 (7) |
| H22 | 1.0257 | 0.0137 | 0.3869 | 0.068* |
| F1 | 0.8929 (3) | −0.36743 (17) | 0.46149 (10) | 0.1008 (7) |
| C20 | 0.8560 (4) | −0.2447 (3) | 0.44071 (14) | 0.0665 (8) |
| C2 | 0.5964 (4) | 0.4062 (3) | 0.42280 (13) | 0.0663 (9) |
| H2A | 0.6725 | 0.4758 | 0.4117 | 0.080* |
| H2B | 0.6451 | 0.3541 | 0.4530 | 0.080* |
| C13 | 0.1585 (4) | 0.8535 (3) | 0.27722 (11) | 0.0645 (8) |
| H13A | 0.2345 | 0.9220 | 0.2882 | 0.097* |
| H13B | 0.0511 | 0.8762 | 0.2907 | 0.097* |
| H13C | 0.1564 | 0.8463 | 0.2367 | 0.097* |
| C11 | 0.3138 (3) | 0.3591 (3) | 0.42485 (11) | 0.0518 (7) |
| C15 | 0.2899 (4) | 0.1980 (3) | 0.34125 (13) | 0.0591 (7) |
| H15A | 0.2996 | 0.1277 | 0.3687 | 0.089* |

| | | | | |
|---|---|---|---|---|
| H15B | 0.3469 | 0.1728 | 0.3074 | 0.089* |
| H15C | 0.1764 | 0.2126 | 0.3327 | 0.089* |
| C14 | 0.3756 (4) | 0.4322 (3) | 0.26721 (11) | 0.0636 (8) |
| H14A | 0.3711 | 0.5156 | 0.2472 | 0.095* |
| H14B | 0.3035 | 0.3690 | 0.2495 | 0.095* |
| H14C | 0.4855 | 0.3983 | 0.2665 | 0.095* |
| C1 | 0.5533 (3) | 0.3168 (3) | 0.37240 (12) | 0.0549 (7) |
| H1 | 0.6103 | 0.3463 | 0.3381 | 0.066* |
| C3 | 0.4327 (4) | 0.4666 (3) | 0.44166 (11) | 0.0614 (8) |
| H3 | 0.4312 | 0.4802 | 0.4827 | 0.074* |
| C12 | 0.1571 (4) | 0.3327 (3) | 0.45174 (12) | 0.0694 (8) |
| H12A | 0.1204 | 0.3945 | 0.4807 | 0.083* |
| H12B | 0.0706 | 0.2946 | 0.4287 | 0.083* |
| C19 | 0.6964 (4) | −0.2039 (3) | 0.44136 (14) | 0.0680 (9) |
| H19 | 0.6145 | −0.2596 | 0.4552 | 0.082* |
| C21 | 0.9811 (4) | −0.1672 (3) | 0.42114 (13) | 0.0651 (8) |
| H21 | 1.0887 | −0.1979 | 0.4215 | 0.078* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O3 | 0.0414 (10) | 0.0521 (10) | 0.0836 (13) | −0.0025 (9) | 0.0026 (10) | 0.0180 (10) |
| O1 | 0.0717 (13) | 0.0486 (9) | 0.0453 (9) | −0.0096 (10) | −0.0136 (9) | 0.0011 (8) |
| C9 | 0.0439 (15) | 0.0497 (14) | 0.0408 (12) | −0.0047 (12) | −0.0050 (11) | 0.0007 (11) |
| C10 | 0.0418 (14) | 0.0432 (13) | 0.0505 (13) | −0.0070 (12) | −0.0056 (11) | 0.0035 (11) |
| C4 | 0.0462 (15) | 0.0498 (13) | 0.0441 (12) | −0.0082 (13) | −0.0059 (11) | 0.0058 (11) |
| O4 | 0.0477 (12) | 0.0872 (16) | 0.0958 (15) | −0.0058 (12) | 0.0064 (11) | 0.0384 (13) |
| C17 | 0.0456 (15) | 0.0544 (15) | 0.0443 (12) | −0.0032 (13) | −0.0004 (11) | −0.0004 (12) |
| C6 | 0.0571 (17) | 0.0521 (16) | 0.0457 (13) | 0.0041 (14) | −0.0044 (13) | 0.0033 (12) |
| C8 | 0.0462 (15) | 0.0538 (15) | 0.0587 (15) | −0.0060 (13) | −0.0093 (13) | −0.0004 (13) |
| C5 | 0.0588 (17) | 0.0450 (14) | 0.0505 (13) | −0.0086 (13) | −0.0078 (13) | 0.0083 (11) |
| C18 | 0.0453 (16) | 0.0545 (16) | 0.0729 (17) | −0.0071 (14) | −0.0045 (14) | 0.0025 (14) |
| O2 | 0.0814 (15) | 0.0665 (12) | 0.0651 (12) | −0.0101 (12) | 0.0039 (11) | 0.0243 (11) |
| C7 | 0.0522 (17) | 0.0653 (17) | 0.0647 (16) | 0.0015 (15) | −0.0151 (14) | 0.0030 (14) |
| C16 | 0.0431 (15) | 0.0613 (16) | 0.0518 (14) | −0.0041 (13) | −0.0025 (12) | 0.0086 (13) |
| C22 | 0.0476 (16) | 0.0649 (18) | 0.0568 (15) | −0.0010 (14) | 0.0076 (13) | 0.0007 (14) |
| F1 | 0.0951 (15) | 0.0492 (9) | 0.1582 (19) | 0.0028 (11) | −0.0263 (15) | 0.0116 (11) |
| C20 | 0.068 (2) | 0.0427 (14) | 0.089 (2) | 0.0002 (15) | −0.0128 (17) | −0.0025 (15) |
| C2 | 0.066 (2) | 0.0576 (16) | 0.0751 (18) | −0.0158 (16) | −0.0304 (16) | 0.0173 (15) |
| C13 | 0.075 (2) | 0.0609 (17) | 0.0571 (15) | 0.0136 (17) | −0.0039 (15) | 0.0045 (14) |
| C11 | 0.0595 (17) | 0.0507 (15) | 0.0453 (13) | −0.0089 (14) | −0.0027 (12) | 0.0101 (12) |
| C15 | 0.0565 (17) | 0.0485 (15) | 0.0723 (18) | −0.0081 (14) | −0.0036 (15) | −0.0055 (14) |
| C14 | 0.0688 (19) | 0.0741 (19) | 0.0481 (14) | 0.0076 (18) | −0.0015 (14) | −0.0008 (14) |
| C1 | 0.0460 (15) | 0.0491 (14) | 0.0697 (16) | −0.0067 (13) | −0.0063 (14) | 0.0159 (14) |
| C3 | 0.085 (2) | 0.0547 (15) | 0.0449 (13) | −0.0137 (17) | −0.0199 (15) | 0.0086 (12) |
| C12 | 0.072 (2) | 0.073 (2) | 0.0625 (17) | −0.0079 (19) | 0.0111 (16) | 0.0079 (16) |
| C19 | 0.062 (2) | 0.0494 (16) | 0.093 (2) | −0.0131 (15) | −0.0047 (17) | 0.0045 (15) |
| C21 | 0.0585 (18) | 0.0628 (18) | 0.0739 (18) | 0.0097 (16) | 0.0012 (15) | −0.0068 (16) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O3—C16 | 1.337 (3) | C7—H7A | 0.9700 |
| O3—C1 | 1.466 (3) | C7—H7B | 0.9700 |
| O1—C3 | 1.432 (3) | C22—C21 | 1.386 (4) |
| O1—C4 | 1.435 (3) | C22—H22 | 0.9300 |
| C9—C14 | 1.537 (4) | F1—C20 | 1.356 (3) |
| C9—C4 | 1.536 (3) | C20—C21 | 1.364 (4) |
| C9—C8 | 1.537 (4) | C20—C19 | 1.365 (5) |
| C9—C10 | 1.576 (3) | C2—C1 | 1.531 (4) |
| C10—C11 | 1.513 (4) | C2—C3 | 1.533 (5) |
| C10—C15 | 1.525 (3) | C2—H2A | 0.9700 |
| C10—C1 | 1.555 (4) | C2—H2B | 0.9700 |
| C4—C5 | 1.501 (3) | C13—H13A | 0.9600 |
| C4—H4 | 0.9800 | C13—H13B | 0.9600 |
| O4—C16 | 1.207 (3) | C13—H13C | 0.9600 |
| C17—C18 | 1.384 (4) | C11—C12 | 1.452 (4) |
| C17—C22 | 1.388 (4) | C11—C3 | 1.502 (4) |
| C17—C16 | 1.480 (4) | C15—H15A | 0.9600 |
| C6—C5 | 1.319 (4) | C15—H15B | 0.9600 |
| C6—C7 | 1.496 (4) | C15—H15C | 0.9600 |
| C6—C13 | 1.501 (4) | C14—H14A | 0.9600 |
| C8—C7 | 1.522 (4) | C14—H14B | 0.9600 |
| C8—H8A | 0.9700 | C14—H14C | 0.9600 |
| C8—H8B | 0.9700 | C1—H1 | 0.9800 |
| C5—H5 | 0.9300 | C3—H3 | 0.9800 |
| C18—C19 | 1.384 (4) | C12—H12A | 0.9700 |
| C18—H18 | 0.9300 | C12—H12B | 0.9700 |
| O2—C11 | 1.445 (3) | C19—H19 | 0.9300 |
| O2—C12 | 1.452 (4) | C21—H21 | 0.9300 |
| | | | |
| C16—O3—C1 | 116.3 (2) | C1—C2—C3 | 104.8 (2) |
| C3—O1—C4 | 112.6 (2) | C1—C2—H2A | 110.8 |
| C14—C9—C4 | 109.0 (2) | C3—C2—H2A | 110.8 |
| C14—C9—C8 | 109.9 (2) | C1—C2—H2B | 110.8 |
| C4—C9—C8 | 106.4 (2) | C3—C2—H2B | 110.8 |
| C14—C9—C10 | 109.6 (2) | H2A—C2—H2B | 108.9 |
| C4—C9—C10 | 110.21 (18) | C6—C13—H13A | 109.5 |
| C8—C9—C10 | 111.7 (2) | C6—C13—H13B | 109.5 |
| C11—C10—C15 | 114.7 (2) | H13A—C13—H13B | 109.5 |
| C11—C10—C1 | 100.2 (2) | C6—C13—H13C | 109.5 |
| C15—C10—C1 | 112.7 (2) | H13A—C13—H13C | 109.5 |
| C11—C10—C9 | 105.7 (2) | H13B—C13—H13C | 109.5 |
| C15—C10—C9 | 113.3 (2) | O2—C11—C12 | 60.15 (17) |
| C1—C10—C9 | 109.2 (2) | O2—C11—C3 | 115.4 (2) |
| O1—C4—C5 | 105.6 (2) | C12—C11—C3 | 125.7 (3) |
| O1—C4—C9 | 111.87 (19) | O2—C11—C10 | 117.6 (2) |
| C5—C4—C9 | 113.6 (2) | C12—C11—C10 | 127.7 (2) |

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| O1—C4—H4 | 108.6 | C3—C11—C10 | 103.1 (2) |
| C5—C4—H4 | 108.6 | C10—C15—H15A | 109.5 |
| C9—C4—H4 | 108.6 | C10—C15—H15B | 109.5 |
| C18—C17—C22 | 119.3 (3) | H15A—C15—H15B | 109.5 |
| C18—C17—C16 | 122.4 (3) | C10—C15—H15C | 109.5 |
| C22—C17—C16 | 118.3 (2) | H15A—C15—H15C | 109.5 |
| C5—C6—C7 | 121.2 (2) | H15B—C15—H15C | 109.5 |
| C5—C6—C13 | 122.4 (3) | C9—C14—H14A | 109.5 |
| C7—C6—C13 | 116.4 (2) | C9—C14—H14B | 109.5 |
| C7—C8—C9 | 112.1 (2) | H14A—C14—H14B | 109.5 |
| C7—C8—H8A | 109.2 | C9—C14—H14C | 109.5 |
| C9—C8—H8A | 109.2 | H14A—C14—H14C | 109.5 |
| C7—C8—H8B | 109.2 | H14B—C14—H14C | 109.5 |
| C9—C8—H8B | 109.2 | O3—C1—C2 | 109.7 (2) |
| H8A—C8—H8B | 107.9 | O3—C1—C10 | 107.5 (2) |
| C6—C5—C4 | 124.3 (2) | C2—C1—C10 | 106.4 (2) |
| C6—C5—H5 | 117.9 | O3—C1—H1 | 111.0 |
| C4—C5—H5 | 117.9 | C2—C1—H1 | 111.0 |
| C19—C18—C17 | 120.2 (3) | C10—C1—H1 | 111.0 |
| C19—C18—H18 | 119.9 | O1—C3—C11 | 109.4 (2) |
| C17—C18—H18 | 119.9 | O1—C3—C2 | 113.3 (2) |
| C11—O2—C12 | 60.18 (17) | C11—C3—C2 | 101.8 (2) |
| C6—C7—C8 | 113.3 (2) | O1—C3—H3 | 110.7 |
| C6—C7—H7A | 108.9 | C11—C3—H3 | 110.7 |
| C8—C7—H7A | 108.9 | C2—C3—H3 | 110.7 |
| C6—C7—H7B | 108.9 | O2—C12—C11 | 59.67 (18) |
| C8—C7—H7B | 108.9 | O2—C12—H12A | 117.8 |
| H7A—C7—H7B | 107.7 | C11—C12—H12A | 117.8 |
| O4—C16—O3 | 123.2 (3) | O2—C12—H12B | 117.8 |
| O4—C16—C17 | 124.2 (3) | C11—C12—H12B | 117.8 |
| O3—C16—C17 | 112.6 (2) | H12A—C12—H12B | 114.9 |
| C21—C22—C17 | 120.9 (3) | C20—C19—C18 | 118.5 (3) |
| C21—C22—H22 | 119.6 | C20—C19—H19 | 120.7 |
| C17—C22—H22 | 119.6 | C18—C19—H19 | 120.7 |
| F1—C20—C21 | 118.1 (3) | C20—C21—C22 | 117.8 (3) |
| F1—C20—C19 | 118.6 (3) | C20—C21—H21 | 121.1 |
| C21—C20—C19 | 123.3 (3) | C22—C21—H21 | 121.1 |

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| C14—C9—C10—C11 | 177.8 (2) | C12—O2—C11—C10 | −119.7 (3) |
| C4—C9—C10—C11 | 57.9 (3) | C15—C10—C11—O2 | 38.5 (3) |
| C8—C9—C10—C11 | −60.2 (3) | C1—C10—C11—O2 | −82.5 (3) |
| C14—C9—C10—C15 | −55.8 (3) | C9—C10—C11—O2 | 164.1 (2) |
| C4—C9—C10—C15 | −175.7 (2) | C15—C10—C11—C12 | −33.7 (4) |
| C8—C9—C10—C15 | 66.2 (3) | C1—C10—C11—C12 | −154.7 (3) |
| C14—C9—C10—C1 | 70.8 (3) | C9—C10—C11—C12 | 91.9 (3) |
| C4—C9—C10—C1 | −49.2 (3) | C15—C10—C11—C3 | 166.8 (2) |
| C8—C9—C10—C1 | −167.2 (2) | C1—C10—C11—C3 | 45.8 (2) |
| C3—O1—C4—C5 | 175.3 (2) | C9—C10—C11—C3 | −67.6 (2) |

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| C3—O1—C4—C9 | 51.3 (3) | C16—O3—C1—C2 | −81.3 (3) |
| C14—C9—C4—O1 | −168.6 (2) | C16—O3—C1—C10 | 163.5 (2) |
| C8—C9—C4—O1 | 72.9 (3) | C3—C2—C1—O3 | −117.3 (2) |
| C10—C9—C4—O1 | −48.3 (3) | C3—C2—C1—C10 | −1.4 (3) |
| C14—C9—C4—C5 | 72.0 (3) | C11—C10—C1—O3 | 90.8 (2) |
| C8—C9—C4—C5 | −46.5 (3) | C15—C10—C1—O3 | −31.6 (3) |
| C10—C9—C4—C5 | −167.7 (2) | C9—C10—C1—O3 | −158.49 (19) |
| C14—C9—C8—C7 | −56.3 (3) | C11—C10—C1—C2 | −26.7 (3) |
| C4—C9—C8—C7 | 61.5 (3) | C15—C10—C1—C2 | −149.1 (2) |
| C10—C9—C8—C7 | −178.2 (2) | C9—C10—C1—C2 | 84.0 (2) |
| C7—C6—C5—C4 | 1.4 (4) | C4—O1—C3—C11 | −64.5 (3) |
| C13—C6—C5—C4 | −177.5 (3) | C4—O1—C3—C2 | 48.2 (3) |
| O1—C4—C5—C6 | −105.8 (3) | O2—C11—C3—O1 | −157.9 (2) |
| C9—C4—C5—C6 | 17.1 (4) | C12—C11—C3—O1 | −87.6 (3) |
| C22—C17—C18—C19 | −0.2 (4) | C10—C11—C3—O1 | 72.5 (3) |
| C16—C17—C18—C19 | −178.3 (3) | O2—C11—C3—C2 | 82.0 (3) |
| C5—C6—C7—C8 | 12.8 (4) | C12—C11—C3—C2 | 152.3 (3) |
| C13—C6—C7—C8 | −168.2 (2) | C10—C11—C3—C2 | −47.6 (2) |
| C9—C8—C7—C6 | −45.5 (3) | C1—C2—C3—O1 | −88.0 (2) |
| C1—O3—C16—O4 | −6.6 (4) | C1—C2—C3—C11 | 29.3 (3) |
| C1—O3—C16—C17 | 173.1 (2) | C3—C11—C12—O2 | −101.4 (3) |
| C18—C17—C16—O4 | −177.1 (3) | C10—C11—C12—O2 | 103.4 (3) |
| C22—C17—C16—O4 | 4.7 (4) | F1—C20—C19—C18 | 179.0 (3) |
| C18—C17—C16—O3 | 3.3 (4) | C21—C20—C19—C18 | 0.3 (5) |
| C22—C17—C16—O3 | −174.9 (2) | C17—C18—C19—C20 | 0.0 (5) |
| C18—C17—C22—C21 | 0.0 (4) | F1—C20—C21—C22 | −179.1 (3) |
| C16—C17—C22—C21 | 178.2 (2) | C19—C20—C21—C22 | −0.4 (5) |
| C12—O2—C11—C3 | 118.1 (3) | C17—C22—C21—C20 | 0.3 (4) |