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# Eplerenone ethanol solvate

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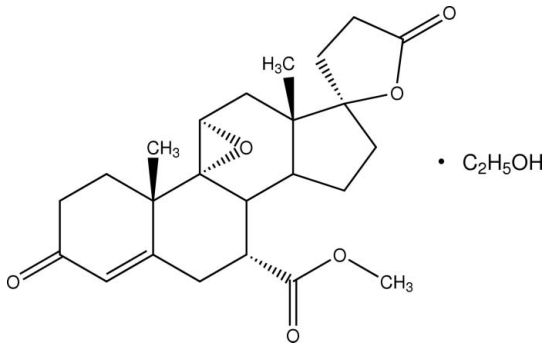
Key indicators: single-crystal X-ray study; $T$ = 295 K; mean $\sigma$(C–C) = 0.005 Å; disorder in solvent or counterion; $R$ factor = 0.056; $wR$ factor = 0.152; data-to-parameter ratio = 8.7.

Eplerenone [systematic name: 7$\alpha$-(methoxycarbonyl)-3-oxo-9$\alpha$,11-epoxy-17$\alpha$-pregn-4-ene-21,17-carbolactone], an aldosterone receptor antagonist, crystallizes from ethanol as a monosolvate, $C_{24}H_{30}O_6\cdot C_2H_6O$. The eplerenone molecule has two five-membered rings, three six-membered rings and one three-membered ring. Both five-membered rings display envelope conformations, while the three six-membered rings assume envelope (cyclohexene), half-chair (cyclohexane sharing one edge with epoxy) and chair (other cyclohexane) conformations. The solvent molecule is disordered equally over two sites. It is linked to the eplerenone molecule by hydrogen bonds.

## Related literature

For background literature, see: Grob *et al.* (1985). For related structures, see: Grob *et al.* (1997); Yang *et al.* (2007); Xu *et al.* (2007). For ring analysis, see: Spek (2003).




## Experimental

### Crystal data

$C_{24}H_{30}O_6\cdot C_2H_6O$
$M_r$ = 460.55
Orthorhombic, $P2_12_12_1$
$a$ = 8.3236 (5) Å
$b$ = 12.8306 (9) Å
$c$ = 23.3173 (13) Å
$V$ = 2490.2 (3) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 295 (2) K
0.20 × 0.16 × 0.14 mm

### Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: none
19548 measured reflections
2559 independent reflections
1955 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.053

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.151
$S$ = 1.05
2559 reflections
293 parameters
6 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.18 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O8—H8$A$···O1$^{i}$ | 0.96 | 2.19 | 3.15 (4) | 178 |
| O9—H9$A$···O1$^{i}$ | 0.98 | 2.34 | 3.32 (4) | 177 |

Symmetry code: (i) $x+\frac{1}{2}, -y+\frac{3}{2}, -z$.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); 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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2439).

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# supporting information

*Acta Cryst.* (2008). E**64**, o829 [doi:10.1107/S1600536808009240]

## Eplerenone ethanol solvate

**Qian Yang, Wei-Dong Ye, Jian-Yong Yuan, Jing-Jing Nie and Duan-Jun Xu**

### S1. Comment

The eplerenone is known as an aldosterone receptor antagonist and can be administered in a therapeutically effective amount where use of an aldosterone receptor antagonist (Grob *et al.*, 1985). The crystal structure of the eplerenone ethanol solvate is reported here.

The crystal of the title compound consists of eplerenone molecules and lattice ethanol molecules (Fig. 1). The molecule of eplerenone contains three six-membered rings, two five-membered rings and one three-membered ring. A ring analysis (Spek, 2003) indicates that three six-membered rings assume different conformations: chair, half-chair and envelope; both five-membered rings display the similar envelope configuration. This agrees with those found in the structure of eplerenone THF solvate (Yang *et al.* 2007) and in the structure of eplerenone dioxane solvate (Xu *et al.*, 2007). The C2—C3 bond distance of 1.343 (6) Å indicates the typical C?C double bond. The C23-ester group forms an intra-molecular C—H···O hydrogen bond with the adjacent C14-methine group (Table 1). This structural feature is also found in the crystal structure of eplerenone dichloromethane solvate (Grob *et al.*, 1997).

In the crystal structure, lattice solvent molecules are disorderly located in the cavities formed by eplerenone molecules and link with eplerenone molecules *via* O—H···O and C—H···O hydrogen bonding (Table 1).

### S2. Experimental

A microcrystalline powder sample of eplerenone was prepared in the manner reported by Grob *et al.* (1997). Single crystals of the title compound were obtained from an ethanol solution of eplerenone.

### S3. Refinement

The lattice ethanol molecule is disordered in the crystal structure; a two-site model with each 0.5 site occupancies was adopted in the refinement. The C—C and C—O distances for the disordered solvent molecule were constrained to 1.50±0.01 and 1.40±0.01 Å, respectively; atomic displacement parameters for non-H atoms of the disordered solvent molecule were constrained to be the same. Hydroxyl H atoms were placed in chemical sensible positions and refined in riding mode with $U_{iso}$(H) = 1.5$U_{eq}$(O). Other H atoms were placed in calculated positions with C—H = 0.93 to 0.98 Å, and refined in riding mode with $U_{iso}$(H) = 1.5$U_{eq}$(C) for methyl or 1.2$U_{eq}$(C) for others. In the absence of significant anomalous scattering effects, Friedel pairs were merged; the absolute configuration was not determined.

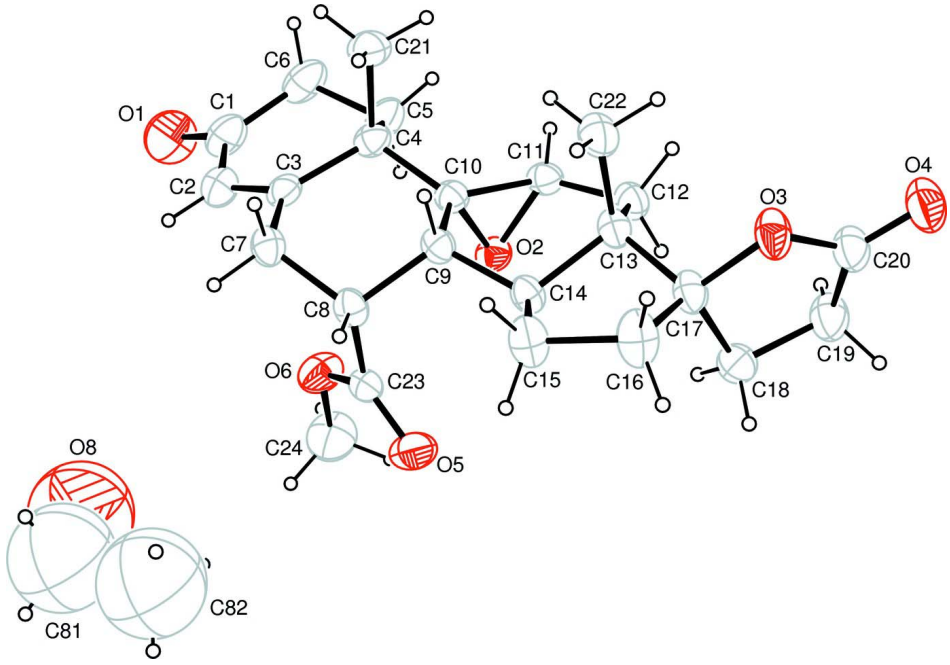


**Figure 1**

The molecular structure of the title compound with 30% probability displacement (arbitrary spheres for H atoms). One of disordered solvent components has been omitted for clarity.

### 7*α*-(methoxycarbonyl)-3-oxo-9*α*,11-epoxy-17*α*-pregn-4-ene-21,17-carbolactone ethanol solvate

*Crystal data*

$C_{24}H_{30}O_6 \cdot C_2H_6O$
$M_r = 460.55$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 8.3236\ (5)$ Å
$b = 12.8306\ (9)$ Å
$c = 23.3173\ (13)$ Å
$V = 2490.2\ (3)$ Å$^3$
$Z = 4$
$F(000) = 992$
$D_x = 1.228$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4788 reflections
$\theta = 3.2–25.2°$
$\mu = 0.09$ mm$^{-1}$
$T = 295$ K
Prism, colorless
0.20 × 0.16 × 0.14 mm

*Data collection*

Rigaku R-AXIS RAPID IP diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
19548 measured reflections
2559 independent reflections
1955 reflections with $I > 2\sigma(I)$
$R_{int} = 0.054$
$\theta_{max} = 25.2°$, $\theta_{min} = 3.0°$
$h = -9\rightarrow8$
$k = -15\rightarrow15$
$l = -27\rightarrow27$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.151$
$S = 1.05$
2559 reflections
293 parameters
6 restraints
Primary atom site location: structure-invariant direct methods

| | |
|---|---|
| Secondary atom site location: difference Fourier map | $w = 1/[\sigma^2(F_o^2) + (0.0933P)^2 + 0.2684P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| Hydrogen site location: inferred from neighbouring sites | $(\Delta/\sigma)_{max} = 0.002$ |
| H-atom parameters constrained | $\Delta\rho_{max} = 0.30$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -0.19$ e Å$^{-3}$ |

*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* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| O1 | −0.0903 (5) | 0.5823 (4) | 0.10016 (17) | 0.1137 (15) | |
| O2 | 0.1457 (3) | 0.82438 (19) | 0.29175 (10) | 0.0474 (6) | |
| O3 | 0.4884 (4) | 0.8328 (2) | 0.49749 (10) | 0.0644 (8) | |
| O4 | 0.3946 (5) | 0.8951 (3) | 0.57892 (12) | 0.0842 (10) | |
| O5 | 0.4856 (5) | 0.9197 (3) | 0.22555 (13) | 0.0866 (11) | |
| O6 | 0.3136 (4) | 0.8354 (2) | 0.17081 (12) | 0.0691 (8) | |
| C1 | −0.0244 (6) | 0.5948 (4) | 0.1464 (2) | 0.0742 (13) | |
| C2 | 0.1457 (6) | 0.6130 (3) | 0.15092 (19) | 0.0642 (11) | |
| H2 | 0.2062 | 0.6122 | 0.1174 | 0.077* | |
| C3 | 0.2222 (5) | 0.6310 (3) | 0.20064 (16) | 0.0476 (9) | |
| C4 | 0.1357 (4) | 0.6367 (3) | 0.25779 (16) | 0.0481 (9) | |
| C5 | −0.0438 (4) | 0.6600 (4) | 0.2471 (2) | 0.0653 (11) | |
| H5A | −0.1024 | 0.6499 | 0.2826 | 0.078* | |
| H5B | −0.0552 | 0.7326 | 0.2361 | 0.078* | |
| C6 | −0.1187 (6) | 0.5921 (4) | 0.2008 (2) | 0.0759 (13) | |
| H6A | −0.1249 | 0.5208 | 0.2145 | 0.091* | |
| H6B | −0.2272 | 0.6160 | 0.1933 | 0.091* | |
| C7 | 0.4024 (4) | 0.6423 (3) | 0.20023 (16) | 0.0515 (9) | |
| H7A | 0.4385 | 0.6512 | 0.1610 | 0.062* | |
| H7B | 0.4502 | 0.5787 | 0.2149 | 0.062* | |
| C8 | 0.4618 (4) | 0.7341 (3) | 0.23596 (14) | 0.0465 (9) | |
| H8 | 0.5790 | 0.7285 | 0.2383 | 0.056* | |
| C9 | 0.3961 (4) | 0.7217 (3) | 0.29729 (14) | 0.0432 (8) | |
| H9 | 0.4299 | 0.6526 | 0.3105 | 0.052* | |
| C10 | 0.2131 (4) | 0.7204 (3) | 0.29597 (15) | 0.0438 (8) | |
| C11 | 0.1258 (4) | 0.7665 (3) | 0.34520 (16) | 0.0467 (9) | |
| H11 | 0.0178 | 0.7384 | 0.3516 | 0.056* | |
| C12 | 0.2055 (4) | 0.8119 (3) | 0.39783 (15) | 0.0508 (9) | |
| H12A | 0.1525 | 0.7852 | 0.4318 | 0.061* | |
| H12B | 0.1925 | 0.8871 | 0.3975 | 0.061* | |

| | | | | | |
|---|---|---|---|---|---|
| C13 | 0.3852 (5) | 0.7856 (3) | 0.40082 (14) | 0.0474 (9) | |
| C14 | 0.4600 (4) | 0.7996 (3) | 0.34071 (14) | 0.0451 (9) | |
| H14 | 0.4309 | 0.8694 | 0.3271 | 0.054* | |
| C15 | 0.6431 (5) | 0.7989 (4) | 0.35129 (17) | 0.0646 (12) | |
| H15A | 0.6865 | 0.7293 | 0.3463 | 0.078* | |
| H15B | 0.6972 | 0.8458 | 0.3250 | 0.078* | |
| C16 | 0.6647 (5) | 0.8359 (5) | 0.41383 (17) | 0.0721 (13) | |
| H16A | 0.7153 | 0.7820 | 0.4368 | 0.086* | |
| H16B | 0.7307 | 0.8981 | 0.4153 | 0.086* | |
| C17 | 0.4943 (5) | 0.8590 (3) | 0.43599 (14) | 0.0534 (10) | |
| C18 | 0.4499 (5) | 0.9740 (3) | 0.43479 (17) | 0.0578 (10) | |
| H18A | 0.3843 | 0.9900 | 0.4015 | 0.069* | |
| H18B | 0.5455 | 1.0173 | 0.4339 | 0.069* | |
| C19 | 0.3553 (7) | 0.9913 (4) | 0.49035 (16) | 0.0727 (13) | |
| H19A | 0.3789 | 1.0590 | 0.5069 | 0.087* | |
| H19B | 0.2406 | 0.9858 | 0.4837 | 0.087* | |
| C20 | 0.4135 (6) | 0.9058 (3) | 0.52799 (16) | 0.0629 (11) | |
| C21 | 0.1522 (6) | 0.5303 (3) | 0.28953 (19) | 0.0659 (11) | |
| H21A | 0.2639 | 0.5144 | 0.2949 | 0.099* | |
| H21B | 0.1027 | 0.4765 | 0.2671 | 0.099* | |
| H21C | 0.1002 | 0.5346 | 0.3262 | 0.099* | |
| C22 | 0.4032 (6) | 0.6722 (3) | 0.42265 (17) | 0.0648 (11) | |
| H22A | 0.3698 | 0.6684 | 0.4620 | 0.097* | |
| H22B | 0.5135 | 0.6511 | 0.4196 | 0.097* | |
| H22C | 0.3374 | 0.6268 | 0.3999 | 0.097* | |
| C23 | 0.4237 (5) | 0.8399 (3) | 0.21110 (15) | 0.0503 (9) | |
| C24 | 0.2627 (8) | 0.9320 (4) | 0.1450 (2) | 0.0944 (18) | |
| H24A | 0.2670 | 0.9867 | 0.1731 | 0.142* | |
| H24B | 0.1546 | 0.9249 | 0.1312 | 0.142* | |
| H24C | 0.3326 | 0.9489 | 0.1136 | 0.142* | |
| O8 | 0.547 (6) | 0.823 (3) | 0.0144 (17) | 0.431 (13)* | 0.50 |
| H8A | 0.5024 | 0.8527 | −0.0201 | 0.646* | 0.50 |
| C81 | 0.715 (6) | 0.815 (6) | 0.021 (2) | 0.431 (13)* | 0.50 |
| H81A | 0.7682 | 0.8468 | −0.0114 | 0.517* | 0.50 |
| H81B | 0.7455 | 0.7423 | 0.0227 | 0.517* | 0.50 |
| C82 | 0.769 (7) | 0.868 (5) | 0.075 (2) | 0.431 (13)* | 0.50 |
| H82A | 0.8188 | 0.8181 | 0.1000 | 0.646* | 0.50 |
| H82B | 0.6778 | 0.8983 | 0.0940 | 0.646* | 0.50 |
| H82C | 0.8447 | 0.9219 | 0.0657 | 0.646* | 0.50 |
| O9 | 0.632 (5) | 0.917 (4) | 0.0182 (15) | 0.431 (13)* | 0.50 |
| H9A | 0.5650 | 0.9195 | −0.0163 | 0.646* | 0.50 |
| C91 | 0.625 (7) | 0.824 (4) | 0.050 (3) | 0.431 (13)* | 0.50 |
| H91A | 0.6519 | 0.7660 | 0.0249 | 0.517* | 0.50 |
| H91B | 0.5155 | 0.8136 | 0.0632 | 0.517* | 0.50 |
| C92 | 0.735 (7) | 0.825 (5) | 0.100 (2) | 0.431 (13)* | 0.50 |
| H92A | 0.7614 | 0.7541 | 0.1100 | 0.646* | 0.50 |
| H92B | 0.6839 | 0.8584 | 0.1315 | 0.646* | 0.50 |
| H92C | 0.8316 | 0.8614 | 0.0898 | 0.646* | 0.50 |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.100 (3) | 0.143 (4) | 0.098 (3) | −0.039 (3) | −0.033 (2) | −0.015 (3) |
| O2 | 0.0473 (14) | 0.0399 (13) | 0.0551 (13) | 0.0060 (11) | 0.0018 (12) | 0.0004 (11) |
| O3 | 0.091 (2) | 0.0635 (17) | 0.0388 (13) | 0.0233 (17) | −0.0092 (14) | −0.0007 (13) |
| O4 | 0.127 (3) | 0.079 (2) | 0.0468 (16) | 0.021 (2) | 0.0019 (18) | 0.0003 (15) |
| O5 | 0.114 (3) | 0.072 (2) | 0.0730 (19) | −0.039 (2) | −0.005 (2) | 0.0047 (17) |
| O6 | 0.087 (2) | 0.0521 (17) | 0.0681 (16) | −0.0022 (17) | −0.0233 (16) | 0.0092 (14) |
| C1 | 0.070 (3) | 0.068 (3) | 0.084 (3) | −0.016 (2) | −0.015 (3) | −0.018 (3) |
| C2 | 0.070 (3) | 0.062 (3) | 0.061 (2) | −0.006 (2) | −0.003 (2) | −0.011 (2) |
| C3 | 0.053 (2) | 0.0314 (18) | 0.058 (2) | 0.0005 (16) | 0.0028 (19) | −0.0040 (16) |
| C4 | 0.0421 (18) | 0.041 (2) | 0.062 (2) | 0.0008 (16) | 0.0030 (18) | −0.0031 (16) |
| C5 | 0.045 (2) | 0.060 (3) | 0.091 (3) | −0.001 (2) | −0.001 (2) | −0.023 (2) |
| C6 | 0.053 (2) | 0.063 (3) | 0.112 (4) | −0.009 (2) | −0.008 (3) | −0.023 (3) |
| C7 | 0.052 (2) | 0.054 (2) | 0.0484 (19) | 0.0091 (18) | 0.0050 (18) | −0.0047 (18) |
| C8 | 0.0345 (18) | 0.061 (2) | 0.0443 (19) | 0.0012 (17) | 0.0017 (16) | −0.0036 (17) |
| C9 | 0.0380 (17) | 0.047 (2) | 0.0443 (17) | 0.0067 (15) | 0.0050 (16) | 0.0000 (16) |
| C10 | 0.0453 (18) | 0.0397 (19) | 0.0464 (18) | 0.0024 (16) | 0.0052 (17) | 0.0026 (16) |
| C11 | 0.0408 (19) | 0.042 (2) | 0.057 (2) | 0.0010 (16) | 0.0100 (17) | 0.0014 (17) |
| C12 | 0.054 (2) | 0.052 (2) | 0.0461 (18) | 0.0068 (19) | 0.0109 (18) | −0.0004 (17) |
| C13 | 0.052 (2) | 0.052 (2) | 0.0380 (17) | 0.0077 (17) | 0.0021 (17) | −0.0009 (16) |
| C14 | 0.0380 (18) | 0.058 (2) | 0.0397 (17) | 0.0012 (17) | 0.0022 (15) | 0.0004 (16) |
| C15 | 0.045 (2) | 0.095 (4) | 0.054 (2) | 0.007 (2) | −0.0038 (19) | −0.012 (2) |
| C16 | 0.053 (2) | 0.103 (4) | 0.060 (2) | 0.017 (3) | −0.010 (2) | −0.016 (3) |
| C17 | 0.063 (2) | 0.060 (2) | 0.0365 (17) | 0.008 (2) | −0.0021 (17) | 0.0003 (17) |
| C18 | 0.066 (3) | 0.056 (2) | 0.051 (2) | −0.001 (2) | −0.0043 (19) | 0.0061 (19) |
| C19 | 0.105 (4) | 0.061 (3) | 0.052 (2) | 0.016 (3) | −0.004 (2) | 0.000 (2) |
| C20 | 0.086 (3) | 0.059 (3) | 0.043 (2) | 0.010 (2) | −0.005 (2) | −0.0002 (19) |
| C21 | 0.071 (3) | 0.048 (2) | 0.079 (3) | −0.001 (2) | 0.014 (2) | 0.005 (2) |
| C22 | 0.088 (3) | 0.056 (2) | 0.051 (2) | 0.017 (2) | 0.004 (2) | 0.0080 (19) |
| C23 | 0.053 (2) | 0.055 (2) | 0.0424 (18) | −0.0129 (19) | 0.0082 (17) | −0.0015 (17) |
| C24 | 0.122 (5) | 0.071 (3) | 0.090 (3) | 0.020 (3) | −0.018 (3) | 0.018 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.221 (6) | C14—C15 | 1.545 (5) |
| O2—C10 | 1.451 (4) | C14—H14 | 0.9800 |
| O2—C11 | 1.460 (4) | C15—C16 | 1.544 (6) |
| O3—C20 | 1.331 (5) | C15—H15A | 0.9700 |
| O3—C17 | 1.474 (4) | C15—H15B | 0.9700 |
| O4—C20 | 1.206 (5) | C16—C17 | 1.538 (6) |
| O5—C23 | 1.195 (5) | C16—H16A | 0.9700 |
| O6—C23 | 1.314 (5) | C16—H16B | 0.9700 |
| O6—C24 | 1.441 (5) | C17—C18 | 1.522 (6) |
| C1—C2 | 1.438 (7) | C18—C19 | 1.532 (6) |
| C1—C6 | 1.492 (7) | C18—H18A | 0.9700 |
| C2—C3 | 1.343 (6) | C18—H18B | 0.9700 |

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|---|---|---|---|
| C2—H2 | 0.9300 | C19—C20 | 1.486 (6) |
| C3—C7 | 1.508 (5) | C19—H19A | 0.9700 |
| C3—C4 | 1.516 (5) | C19—H19B | 0.9700 |
| C4—C10 | 1.536 (5) | C21—H21A | 0.9600 |
| C4—C5 | 1.544 (5) | C21—H21B | 0.9600 |
| C4—C21 | 1.559 (5) | C21—H21C | 0.9600 |
| C5—C6 | 1.520 (6) | C22—H22A | 0.9600 |
| C5—H5A | 0.9700 | C22—H22B | 0.9600 |
| C5—H5B | 0.9700 | C22—H22C | 0.9600 |
| C6—H6A | 0.9700 | C24—H24A | 0.9600 |
| C6—H6B | 0.9700 | C24—H24B | 0.9600 |
| C7—C8 | 1.525 (5) | C24—H24C | 0.9600 |
| C7—H7A | 0.9700 | O8—C81 | 1.409 (11) |
| C7—H7B | 0.9700 | O8—H8A | 0.9668 |
| C8—C23 | 1.510 (5) | C81—C82 | 1.494 (11) |
| C8—C9 | 1.539 (5) | C81—H81A | 0.9700 |
| C8—H8 | 0.9800 | C81—H81B | 0.9700 |
| C9—C14 | 1.519 (5) | C82—H82A | 0.9600 |
| C9—C10 | 1.523 (5) | C82—H82B | 0.9600 |
| C9—H9 | 0.9800 | C82—H82C | 0.9600 |
| C10—C11 | 1.482 (5) | O9—C91 | 1.403 (11) |
| C11—C12 | 1.512 (5) | O9—H9A | 0.9804 |
| C11—H11 | 0.9800 | C91—C92 | 1.483 (11) |
| C12—C13 | 1.535 (5) | C91—H91A | 0.9700 |
| C12—H12A | 0.9700 | C91—H91B | 0.9700 |
| C12—H12B | 0.9700 | C92—H92A | 0.9600 |
| C13—C14 | 1.544 (5) | C92—H92B | 0.9600 |
| C13—C17 | 1.545 (6) | C92—H92C | 0.9600 |
| C13—C22 | 1.548 (6) | | |

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| C10—O2—C11 | 61.2 (2) | C14—C15—H15A | 110.7 |
| C20—O3—C17 | 112.0 (3) | C16—C15—H15B | 110.7 |
| C23—O6—C24 | 117.8 (4) | C14—C15—H15B | 110.7 |
| O1—C1—C2 | 121.9 (5) | H15A—C15—H15B | 108.8 |
| O1—C1—C6 | 120.8 (4) | C17—C16—C15 | 105.6 (3) |
| C2—C1—C6 | 117.3 (4) | C17—C16—H16A | 110.6 |
| C3—C2—C1 | 123.9 (4) | C15—C16—H16A | 110.6 |
| C3—C2—H2 | 118.1 | C17—C16—H16B | 110.6 |
| C1—C2—H2 | 118.1 | C15—C16—H16B | 110.6 |
| C2—C3—C7 | 118.9 (4) | H16A—C16—H16B | 108.7 |
| C2—C3—C4 | 122.8 (3) | O3—C17—C18 | 103.4 (3) |
| C7—C3—C4 | 118.3 (3) | O3—C17—C16 | 108.3 (3) |
| C3—C4—C10 | 110.1 (3) | C18—C17—C16 | 113.9 (4) |
| C3—C4—C5 | 109.1 (3) | O3—C17—C13 | 111.0 (3) |
| C10—C4—C5 | 111.4 (3) | C18—C17—C13 | 116.0 (3) |
| C3—C4—C21 | 109.4 (3) | C16—C17—C13 | 104.3 (3) |
| C10—C4—C21 | 107.4 (3) | C17—C18—C19 | 104.4 (3) |
| C5—C4—C21 | 109.4 (3) | C17—C18—H18A | 110.9 |

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| C6—C5—C4 | 113.6 (3) | C19—C18—H18A | 110.9 |
| C6—C5—H5A | 108.8 | C17—C18—H18B | 110.9 |
| C4—C5—H5A | 108.8 | C19—C18—H18B | 110.9 |
| C6—C5—H5B | 108.8 | H18A—C18—H18B | 108.9 |
| C4—C5—H5B | 108.8 | C20—C19—C18 | 103.0 (4) |
| H5A—C5—H5B | 107.7 | C20—C19—H19A | 111.2 |
| C1—C6—C5 | 112.0 (4) | C18—C19—H19A | 111.2 |
| C1—C6—H6A | 109.2 | C20—C19—H19B | 111.2 |
| C5—C6—H6A | 109.2 | C18—C19—H19B | 111.2 |
| C1—C6—H6B | 109.2 | H19A—C19—H19B | 109.1 |
| C5—C6—H6B | 109.2 | O4—C20—O3 | 120.5 (4) |
| H6A—C6—H6B | 107.9 | O4—C20—C19 | 128.5 (4) |
| C3—C7—C8 | 113.1 (3) | O3—C20—C19 | 110.9 (3) |
| C3—C7—H7A | 109.0 | C4—C21—H21A | 109.5 |
| C8—C7—H7A | 109.0 | C4—C21—H21B | 109.5 |
| C3—C7—H7B | 109.0 | H21A—C21—H21B | 109.5 |
| C8—C7—H7B | 109.0 | C4—C21—H21C | 109.5 |
| H7A—C7—H7B | 107.8 | H21A—C21—H21C | 109.5 |
| C23—C8—C7 | 114.6 (3) | H21B—C21—H21C | 109.5 |
| C23—C8—C9 | 112.0 (3) | C13—C22—H22A | 109.5 |
| C7—C8—C9 | 108.2 (3) | C13—C22—H22B | 109.5 |
| C23—C8—H8 | 107.2 | H22A—C22—H22B | 109.5 |
| C7—C8—H8 | 107.2 | C13—C22—H22C | 109.5 |
| C9—C8—H8 | 107.2 | H22A—C22—H22C | 109.5 |
| C14—C9—C10 | 111.8 (3) | H22B—C22—H22C | 109.5 |
| C14—C9—C8 | 115.3 (3) | O5—C23—O6 | 122.7 (4) |
| C10—C9—C8 | 109.7 (3) | O5—C23—C8 | 124.9 (4) |
| C14—C9—H9 | 106.5 | O6—C23—C8 | 112.4 (3) |
| C10—C9—H9 | 106.5 | O6—C24—H24A | 109.5 |
| C8—C9—H9 | 106.5 | O6—C24—H24B | 109.5 |
| O2—C10—C11 | 59.7 (2) | H24A—C24—H24B | 109.5 |
| O2—C10—C9 | 112.2 (3) | O6—C24—H24C | 109.5 |
| C11—C10—C9 | 118.1 (3) | H24A—C24—H24C | 109.5 |
| O2—C10—C4 | 116.2 (3) | H24B—C24—H24C | 109.5 |
| C11—C10—C4 | 121.5 (3) | C81—O8—H8A | 120.4 |
| C9—C10—C4 | 116.0 (3) | C81—O8—H9A | 91.0 |
| O2—C11—C10 | 59.1 (2) | O8—C81—C82 | 111.1 (11) |
| O2—C11—C12 | 116.5 (3) | O8—C81—H81A | 109.4 |
| C10—C11—C12 | 124.6 (3) | C82—C81—H81A | 109.4 |
| O2—C11—H11 | 114.9 | O8—C81—H81B | 109.4 |
| C10—C11—H11 | 114.9 | C82—C81—H81B | 109.4 |
| C12—C11—H11 | 114.9 | H81A—C81—H81B | 108.0 |
| C11—C12—C13 | 112.3 (3) | C81—C82—H82A | 109.5 |
| C11—C12—H12A | 109.1 | C81—C82—H82B | 109.5 |
| C13—C12—H12A | 109.1 | H82A—C82—H82B | 109.5 |
| C11—C12—H12B | 109.1 | C81—C82—H82C | 109.5 |
| C13—C12—H12B | 109.1 | H82A—C82—H82C | 109.5 |
| H12A—C12—H12B | 107.9 | H82B—C82—H82C | 109.5 |

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| C12—C13—C14 | 109.0 (3) | C91—O9—H9A | 115.6 |
| C12—C13—C17 | 117.6 (3) | O9—C91—C92 | 112.3 (12) |
| C14—C13—C17 | 100.0 (3) | O9—C91—H91A | 109.1 |
| C12—C13—C22 | 108.5 (3) | C92—C91—H91A | 109.1 |
| C14—C13—C22 | 111.7 (3) | O9—C91—H91B | 109.1 |
| C17—C13—C22 | 110.0 (3) | C92—C91—H91B | 109.1 |
| C9—C14—C13 | 112.8 (3) | H91A—C91—H91B | 107.9 |
| C9—C14—C15 | 116.6 (3) | C91—C92—H92A | 109.5 |
| C13—C14—C15 | 104.6 (3) | C91—C92—H92B | 109.5 |
| C9—C14—H14 | 107.5 | H92A—C92—H92B | 109.5 |
| C13—C14—H14 | 107.5 | C91—C92—H92C | 109.5 |
| C15—C14—H14 | 107.5 | H92A—C92—H92C | 109.5 |
| C16—C15—C14 | 105.3 (3) | H92B—C92—H92C | 109.5 |
| C16—C15—H15A | 110.7 | | |

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| O1—C1—C2—C3 | 177.4 (5) | O2—C11—C12—C13 | −81.0 (4) |
| C6—C1—C2—C3 | −2.3 (7) | C10—C11—C12—C13 | −11.7 (5) |
| C1—C2—C3—C7 | 176.2 (4) | C11—C12—C13—C14 | 43.1 (4) |
| C1—C2—C3—C4 | −1.5 (7) | C11—C12—C13—C17 | 155.9 (3) |
| C2—C3—C4—C10 | −143.4 (4) | C11—C12—C13—C22 | −78.6 (4) |
| C7—C3—C4—C10 | 38.9 (4) | C10—C9—C14—C13 | 51.8 (4) |
| C2—C3—C4—C5 | −20.9 (5) | C8—C9—C14—C13 | 178.0 (3) |
| C7—C3—C4—C5 | 161.4 (3) | C10—C9—C14—C15 | 172.9 (3) |
| C2—C3—C4—C21 | 98.7 (4) | C8—C9—C14—C15 | −60.9 (5) |
| C7—C3—C4—C21 | −79.0 (4) | C12—C13—C14—C9 | −66.2 (4) |
| C3—C4—C5—C6 | 47.3 (5) | C17—C13—C14—C9 | 169.9 (3) |
| C10—C4—C5—C6 | 169.0 (4) | C22—C13—C14—C9 | 53.7 (4) |
| C21—C4—C5—C6 | −72.4 (5) | C12—C13—C14—C15 | 166.1 (3) |
| O1—C1—C6—C5 | −150.7 (5) | C17—C13—C14—C15 | 42.2 (4) |
| C2—C1—C6—C5 | 29.0 (7) | C22—C13—C14—C15 | −74.1 (4) |
| C4—C5—C6—C1 | −52.5 (6) | C9—C14—C15—C16 | −151.0 (4) |
| C2—C3—C7—C8 | 134.9 (4) | C13—C14—C15—C16 | −25.7 (5) |
| C4—C3—C7—C8 | −47.3 (5) | C14—C15—C16—C17 | −1.4 (5) |
| C3—C7—C8—C23 | −70.2 (4) | C20—O3—C17—C18 | 14.6 (5) |
| C3—C7—C8—C9 | 55.7 (4) | C20—O3—C17—C16 | 135.7 (4) |
| C23—C8—C9—C14 | −59.7 (4) | C20—O3—C17—C13 | −110.5 (4) |
| C7—C8—C9—C14 | 172.9 (3) | C15—C16—C17—O3 | 146.3 (4) |
| C23—C8—C9—C10 | 67.5 (4) | C15—C16—C17—C18 | −99.4 (4) |
| C7—C8—C9—C10 | −59.9 (4) | C15—C16—C17—C13 | 28.0 (5) |
| C11—O2—C10—C9 | −110.5 (3) | C12—C13—C17—O3 | 82.8 (4) |
| C11—O2—C10—C4 | 112.7 (4) | C14—C13—C17—O3 | −159.5 (3) |
| C14—C9—C10—O2 | 48.9 (4) | C22—C13—C17—O3 | −41.9 (4) |
| C8—C9—C10—O2 | −80.2 (4) | C12—C13—C17—C18 | −34.7 (5) |
| C14—C9—C10—C11 | −17.5 (5) | C14—C13—C17—C18 | 83.0 (4) |
| C8—C9—C10—C11 | −146.7 (3) | C22—C13—C17—C18 | −159.5 (3) |
| C14—C9—C10—C4 | −174.2 (3) | C12—C13—C17—C16 | −160.8 (3) |
| C8—C9—C10—C4 | 56.6 (4) | C14—C13—C17—C16 | −43.1 (4) |
| C3—C4—C10—O2 | 91.2 (4) | C22—C13—C17—C16 | 74.5 (4) |

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| C5—C4—C10—O2 | −30.0 (5) | O3—C17—C18—C19 | −23.4 (4) |
| C21—C4—C10—O2 | −149.7 (3) | C16—C17—C18—C19 | −140.6 (4) |
| C3—C4—C10—C11 | 160.2 (3) | C13—C17—C18—C19 | 98.3 (4) |
| C5—C4—C10—C11 | 39.1 (5) | C17—C18—C19—C20 | 23.9 (5) |
| C21—C4—C10—C11 | −80.7 (4) | C17—O3—C20—O4 | 178.1 (4) |
| C3—C4—C10—C9 | −44.0 (4) | C17—O3—C20—C19 | 1.0 (5) |
| C5—C4—C10—C9 | −165.1 (3) | C18—C19—C20—O4 | 167.2 (5) |
| C21—C4—C10—C9 | 75.1 (4) | C18—C19—C20—O3 | −16.1 (5) |
| C10—O2—C11—C12 | 116.1 (3) | C24—O6—C23—O5 | −2.4 (6) |
| C9—C10—C11—O2 | 100.7 (3) | C24—O6—C23—C8 | 177.9 (4) |
| C4—C10—C11—O2 | −104.0 (3) | C7—C8—C23—O5 | −164.2 (4) |
| O2—C10—C11—C12 | −102.7 (4) | C9—C8—C23—O5 | 72.0 (5) |
| C9—C10—C11—C12 | −2.0 (5) | C7—C8—C23—O6 | 15.5 (4) |
| C4—C10—C11—C12 | 153.4 (3) | C9—C8—C23—O6 | −108.3 (3) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O8—H8*A*···O1$^{i}$ | 0.96 | 2.19 | 3.15 (4) | 178 |
| O9—H9*A*···O1$^{i}$ | 0.98 | 2.34 | 3.32 (4) | 177 |
| C6—H6*A*···O2$^{ii}$ | 0.97 | 2.53 | 3.447 (6) | 157 |
| C7—H7*B*···O5$^{iii}$ | 0.97 | 2.52 | 3.467 (5) | 164 |
| C11—H11···O4$^{iv}$ | 0.98 | 2.57 | 3.337 (5) | 135 |
| C14—H14···O5 | 0.98 | 2.50 | 3.103 (5) | 120 |
| C21—H21*A*···O5$^{iii}$ | 0.96 | 2.46 | 3.351 (6) | 154 |
| C92—H92*A*···O1$^{v}$ | 0.96 | 2.54 | 3.44 (6) | 155 |

Symmetry codes: (i) *x*+1/2, −*y*+3/2, −*z*; (ii) −*x*, *y*−1/2, −*z*+1/2; (iii) −*x*+1, *y*−1/2, −*z*+1/2; (iv) *x*−1/2, −*y*+3/2, −*z*+1; (v) *x*+1, *y*, *z*.