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# (1*S**,2*R**,4a*S**,6a*S**,6b*R**,10*S**,12a*R**,14a*S**)-10-Hydroxy-1,2,6a,6b,9,9,12a-heptamethylperhydropicene-4a,14a-carbolactone

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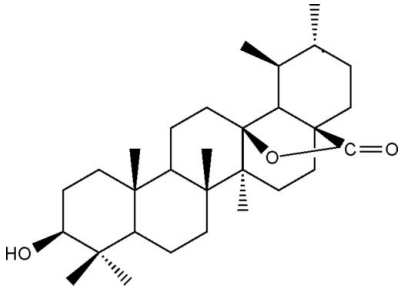
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.052; $wR$ factor = 0.142; data-to-parameter ratio = 8.1.

The title compound, $C_{30}H_{48}O_3$, was extracted from the plant *Dracocephalum rupestre* Hance. The molecule contains five fused cyclohexane rings and one five-membered lactone ring. Intermolecular O—H···O hydrogen bonds between the hydroxyl and carbonyl groups link the molecules into chains along [010]. The absolute structure has not been determined.

## Related literature

For related literature concerning the title compound and the plant *Dracocephalum rupestre* Hance, see: Jiangsu College of New Medicine (1977); Katai *et al.* (1983).




## Experimental

### Crystal data

$C_{30}H_{48}O_3$
$M_r$ = 456.68
Monoclinic, $P2_1$
$a$ = 8.156 (3) Å
$b$ = 12.005 (5) Å
$c$ = 13.475 (5) Å
$\beta$ = 90.520 (7)°
$V$ = 1319.3 (9) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.07 mm$^{-1}$
$T$ = 293 K
0.60 × 0.50 × 0.30 mm

### Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.958, $T_{max}$ = 0.979
5150 measured reflections
2428 independent reflections
2052 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.032

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.142
$S$ = 1.03
2428 reflections
299 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.25 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3$A$···O2$^{i}$ | 0.82 | 2.24 | 3.059 (4) | 176 |

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Financial support from the Science and Technology Commission of Taiyuan (2006), and from the Undergraduate Innovation Item of Shanxi Medical University (No. 200448) is gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2335).

## References

Bruker (2000). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Jiangsu College of New Medicine (1977). *A Dictionary of the Traditional Chinese Medicines*, p. 549. Shanghai: Shanghai Science and Technology Press.
Katai, M., Terai, T. & Meguri, H. (1983). *Chem. Pharm. Bull.* **31**, 1567–1571.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

# supporting information

*Acta Cryst.* (2009). E**65**, o753 [doi:10.1107/S1600536809008253]

## (1*S**,2*R**,4a*S**,6a*S**,6b*R**,10*S**,12a*R**,14a*S**)-10-Hydroxy-1,2,6a,6b,9,9,12a-heptamethylperhydropicene-4a,14a-carbolactone

**Dan-Wei Ou-yang, Jian-Ping Gao, Qing-Shan Li and Jian-Ping Guo**

### S1. Comment

The title compound is extracted from the plant *Dracocephalum rupestre Hance* (Jiangsu College of New Medicine, 1977) with ethanol. The compound (Katai *et al.*, 1983) was successfully crystallized from methanol. There are five six-membered rings and one five-membered ring in the molecule. The six-membered rings are composed of $sp^3$-hybridised C and the five-membered ring is a lactone in which C28 is $sp^2$ hybridised. The bond distances between C28 and O are 1.349 (3) [O1—C28] and 1.218 (4) Å [O2—C28]. The O2—C28 bond length of 1.216 (5) Å is a typical C=O double bond.

### S2. Experimental

The dry aerial part of the plant (5.3 kg) was extracted with 95% ethanol 3 times under reflux. The ethanol extract was diluted with a large amount of water,and then extracted with petroleum ether, chloroform, EtOAc and n-butanol. The chloroform fraction (70 g) was subjected to Si gel column (1.5 kg,200–300 mesh) chromatography eluting with a gradient (petroleum ether-EtOAc, 99:1, 98:2, 97:3, 95:5, 9:1, 8:2,7:3, 1:1, *v*/*v*) to obtain 8 fractions (F1—F8). Fraction F3 (19.4 g) was separated by Si gel column (500 g, 200–300 mesh) chromatography eluting with a gradient (chloroform-methanol, 99:1, 98:2, 97:3, 95:5, 9:1,8:2,7:3, 1:1, *v*/*v*) to yield four portions. Subfraction 1 was subsequently subjected to Si gel column chromatography eluting with chloroform-methanol (10:1), and recrystallized from methanol, to obtain the title compound (25 mg).

### S3. Refinement

H atoms were placed geometrically and allowed to ride with $U_{iso}$(H) = 1.2 or 1.5$U_{eq}$(C/O). In the absence of significant anomalous scattering, Friedel pairs were merged as equivalent data, and the absolute structure has not been determined.

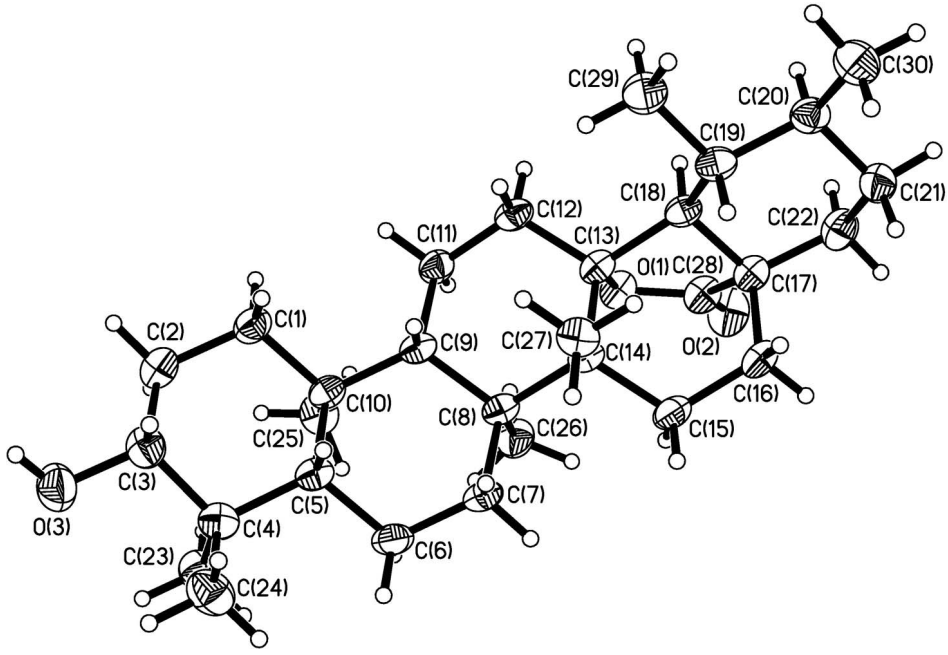



**Figure 1**

Molecular structure showing displacement ellipsoids at 50% probabilty for non-H atoms.

### (1*S**,2*R**,4a*S**,6a*S**,6b*R**,10*S**, 12a*R**,14a*S**)-10-Hydroxy-1,2,6a,6b,9,9,12a-heptamethylperhydropicene-4a,14a-carbolactone

*Crystal data*

$C_{30}H_{48}O_3$
$M_r = 456.68$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 8.156\ (3)$ Å
$b = 12.005\ (5)$ Å
$c = 13.475\ (5)$ Å
$\beta = 90.520\ (7)°$
$V = 1319.3\ (9)$ Å$^3$
$Z = 2$
$F(000) = 504$
$D_x = 1.150$ Mg m$^{-3}$
Melting point: 519 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2717 reflections
$\theta = 2.3–26.2°$
$\mu = 0.07$ mm$^{-1}$
$T = 293$ K
Block, colourless
0.60 × 0.50 × 0.30 mm

*Data collection*

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.958$, $T_{max} = 0.979$
5150 measured reflections
2428 independent reflections
2052 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.5°$
$h = -9\rightarrow7$
$k = -14\rightarrow14$
$l = -13\rightarrow16$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.142$
$S = 1.03$
2428 reflections
299 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0816P)^2 + 0.1773P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å$^{-3}$
$\Delta\rho_{min} = -0.25$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | −0.1317 (3) | 0.74752 (19) | 0.69165 (16) | 0.0521 (6) |
| O2 | −0.1163 (4) | 0.6395 (2) | 0.5579 (2) | 0.0776 (9) |
| O3 | 0.2976 (4) | 0.9857 (3) | 1.2991 (2) | 0.0794 (9) |
| H3A | 0.2459 | 1.0276 | 1.3353 | 0.119* |
| C1 | −0.0162 (4) | 0.9125 (4) | 1.1007 (3) | 0.0566 (9) |
| H1A | −0.0476 | 0.9870 | 1.0801 | 0.068* |
| H1B | −0.1154 | 0.8681 | 1.1051 | 0.068* |
| C2 | 0.0658 (5) | 0.9185 (4) | 1.2036 (3) | 0.0654 (10) |
| H2A | −0.0095 | 0.9529 | 1.2497 | 0.078* |
| H2B | 0.0879 | 0.8435 | 1.2268 | 0.078* |
| C3 | 0.2236 (5) | 0.9837 (3) | 1.2033 (3) | 0.0592 (9) |
| H3B | 0.1969 | 1.0606 | 1.1852 | 0.071* |
| C4 | 0.3498 (4) | 0.9406 (3) | 1.1284 (3) | 0.0543 (8) |
| C5 | 0.2606 (4) | 0.9273 (3) | 1.0255 (2) | 0.0464 (8) |
| H5A | 0.2293 | 1.0034 | 1.0072 | 0.056* |
| C6 | 0.3736 (4) | 0.8899 (4) | 0.9416 (3) | 0.0590 (9) |
| H6A | 0.4776 | 0.9287 | 0.9472 | 0.071* |
| H6B | 0.3946 | 0.8107 | 0.9475 | 0.071* |
| C7 | 0.2955 (4) | 0.9145 (4) | 0.8405 (3) | 0.0583 (9) |
| H7A | 0.3690 | 0.8888 | 0.7891 | 0.070* |
| H7B | 0.2838 | 0.9945 | 0.8332 | 0.070* |
| C8 | 0.1261 (4) | 0.8596 (3) | 0.8242 (2) | 0.0466 (8) |
| C9 | 0.0174 (3) | 0.8830 (3) | 0.9165 (2) | 0.0432 (7) |
| H9A | −0.0032 | 0.9634 | 0.9147 | 0.052* |
| C10 | 0.0967 (4) | 0.8611 (3) | 1.0212 (2) | 0.0457 (7) |

| | | | | |
|---|---|---|---|---|
| C11 | −0.1523 (4) | 0.8290 (3) | 0.9008 (2) | 0.0510 (8) |
| H11A | −0.1393 | 0.7492 | 0.8930 | 0.061* |
| H11B | −0.2190 | 0.8421 | 0.9589 | 0.061* |
| C12 | −0.2392 (4) | 0.8762 (3) | 0.8096 (2) | 0.0527 (8) |
| H12A | −0.3415 | 0.8363 | 0.7998 | 0.063* |
| H12B | −0.2660 | 0.9536 | 0.8222 | 0.063* |
| C13 | −0.1418 (4) | 0.8697 (3) | 0.7150 (2) | 0.0439 (7) |
| C14 | 0.0382 (4) | 0.9125 (3) | 0.7273 (2) | 0.0430 (7) |
| C15 | 0.1341 (4) | 0.8817 (4) | 0.6322 (2) | 0.0550 (9) |
| H15A | 0.2303 | 0.9289 | 0.6284 | 0.066* |
| H15B | 0.1718 | 0.8053 | 0.6382 | 0.066* |
| C16 | 0.0360 (4) | 0.8931 (3) | 0.5351 (2) | 0.0532 (9) |
| H16A | 0.0952 | 0.8559 | 0.4825 | 0.064* |
| H16B | 0.0282 | 0.9714 | 0.5179 | 0.064* |
| C17 | −0.1378 (4) | 0.8442 (3) | 0.5403 (2) | 0.0477 (8) |
| C18 | −0.2370 (4) | 0.9073 (3) | 0.6187 (2) | 0.0445 (7) |
| H18A | −0.3417 | 0.8675 | 0.6216 | 0.053* |
| C19 | −0.2835 (4) | 1.0265 (3) | 0.5865 (2) | 0.0496 (8) |
| H19A | −0.1825 | 1.0694 | 0.5774 | 0.059* |
| C20 | −0.3750 (4) | 1.0192 (3) | 0.4849 (2) | 0.0530 (8) |
| H20A | −0.4761 | 0.9767 | 0.4949 | 0.064* |
| C21 | −0.2730 (5) | 0.9578 (4) | 0.4078 (3) | 0.0577 (9) |
| H21A | −0.1745 | 1.0005 | 0.3949 | 0.069* |
| H21B | −0.3353 | 0.9533 | 0.3462 | 0.069* |
| C22 | −0.2241 (5) | 0.8403 (3) | 0.4399 (3) | 0.0592 (9) |
| H22A | −0.3210 | 0.7939 | 0.4441 | 0.071* |
| H22B | −0.1516 | 0.8080 | 0.3910 | 0.071* |
| C23 | 0.4318 (5) | 0.8333 (4) | 1.1653 (3) | 0.0712 (11) |
| H23A | 0.4847 | 0.8471 | 1.2281 | 0.107* |
| H23B | 0.5118 | 0.8094 | 1.1181 | 0.107* |
| H23C | 0.3504 | 0.7763 | 1.1730 | 0.107* |
| C24 | 0.4838 (5) | 1.0301 (4) | 1.1204 (3) | 0.0748 (12) |
| H24A | 0.5386 | 1.0381 | 1.1833 | 0.112* |
| H24B | 0.4348 | 1.0998 | 1.1018 | 0.112* |
| H24C | 0.5616 | 1.0083 | 1.0710 | 0.112* |
| C25 | 0.1145 (5) | 0.7347 (3) | 1.0452 (3) | 0.0612 (9) |
| H25A | 0.1114 | 0.7239 | 1.1157 | 0.092* |
| H25B | 0.2171 | 0.7080 | 1.0202 | 0.092* |
| H25C | 0.0261 | 0.6944 | 1.0144 | 0.092* |
| C26 | 0.1588 (5) | 0.7321 (3) | 0.8123 (3) | 0.0622 (10) |
| H26A | 0.2380 | 0.7085 | 0.8611 | 0.093* |
| H26B | 0.2002 | 0.7176 | 0.7471 | 0.093* |
| H26C | 0.0584 | 0.6918 | 0.8214 | 0.093* |
| C27 | 0.0327 (4) | 1.0412 (3) | 0.7341 (3) | 0.0542 (8) |
| H27A | −0.0192 | 1.0708 | 0.6755 | 0.081* |
| H27B | 0.1423 | 1.0698 | 0.7395 | 0.081* |
| H27C | −0.0286 | 1.0629 | 0.7914 | 0.081* |
| C28 | −0.1268 (4) | 0.7329 (3) | 0.5923 (3) | 0.0546 (8) |

| | | | | |
|---|---|---|---|---|
| C29 | −0.3888 (5) | 1.0861 (4) | 0.6629 (3) | 0.0715 (11) |
| H29A | −0.3304 | 1.0900 | 0.7250 | 0.107* |
| H29B | −0.4892 | 1.0456 | 0.6718 | 0.107* |
| H29C | −0.4132 | 1.1600 | 0.6401 | 0.107* |
| C30 | −0.4225 (5) | 1.1339 (4) | 0.4454 (3) | 0.0727 (11) |
| H30A | −0.4788 | 1.1258 | 0.3830 | 0.109* |
| H30B | −0.3254 | 1.1778 | 0.4363 | 0.109* |
| H30C | −0.4930 | 1.1702 | 0.4919 | 0.109* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0634 (14) | 0.0347 (12) | 0.0582 (13) | −0.0028 (11) | 0.0101 (10) | 0.0014 (10) |
| O2 | 0.115 (2) | 0.0379 (14) | 0.0798 (18) | 0.0061 (15) | 0.0098 (16) | −0.0079 (13) |
| O3 | 0.088 (2) | 0.084 (2) | 0.0662 (16) | 0.0096 (17) | −0.0086 (15) | −0.0169 (15) |
| C1 | 0.0500 (19) | 0.064 (2) | 0.056 (2) | −0.0025 (17) | 0.0118 (15) | −0.0011 (17) |
| C2 | 0.067 (2) | 0.072 (3) | 0.057 (2) | 0.002 (2) | 0.0129 (18) | −0.0041 (18) |
| C3 | 0.066 (2) | 0.051 (2) | 0.060 (2) | 0.0044 (18) | −0.0036 (17) | −0.0040 (16) |
| C4 | 0.0513 (19) | 0.049 (2) | 0.063 (2) | 0.0008 (16) | −0.0003 (16) | 0.0036 (16) |
| C5 | 0.0414 (16) | 0.0422 (18) | 0.0556 (19) | 0.0017 (14) | 0.0075 (14) | 0.0048 (14) |
| C6 | 0.0375 (16) | 0.072 (3) | 0.068 (2) | 0.0045 (17) | 0.0072 (15) | 0.0029 (19) |
| C7 | 0.0363 (16) | 0.079 (3) | 0.060 (2) | 0.0016 (17) | 0.0129 (14) | 0.0043 (18) |
| C8 | 0.0405 (16) | 0.0441 (18) | 0.0554 (18) | 0.0041 (14) | 0.0097 (14) | −0.0002 (14) |
| C9 | 0.0378 (15) | 0.0371 (17) | 0.0548 (18) | −0.0002 (13) | 0.0093 (13) | 0.0008 (14) |
| C10 | 0.0448 (16) | 0.0393 (17) | 0.0534 (18) | −0.0012 (14) | 0.0095 (13) | 0.0012 (14) |
| C11 | 0.0457 (17) | 0.057 (2) | 0.0503 (18) | −0.0076 (16) | 0.0087 (14) | 0.0028 (15) |
| C12 | 0.0392 (16) | 0.059 (2) | 0.060 (2) | −0.0053 (16) | 0.0118 (14) | −0.0015 (17) |
| C13 | 0.0466 (16) | 0.0351 (16) | 0.0503 (17) | −0.0012 (13) | 0.0116 (13) | −0.0015 (13) |
| C14 | 0.0369 (15) | 0.0408 (17) | 0.0514 (18) | 0.0015 (13) | 0.0093 (13) | −0.0015 (13) |
| C15 | 0.0449 (17) | 0.063 (2) | 0.058 (2) | 0.0042 (17) | 0.0160 (15) | 0.0022 (17) |
| C16 | 0.0548 (19) | 0.053 (2) | 0.052 (2) | −0.0003 (16) | 0.0198 (15) | 0.0009 (16) |
| C17 | 0.0532 (18) | 0.0382 (17) | 0.0520 (18) | −0.0007 (14) | 0.0071 (15) | −0.0029 (14) |
| C18 | 0.0388 (15) | 0.0405 (17) | 0.0544 (19) | −0.0043 (13) | 0.0078 (14) | −0.0005 (14) |
| C19 | 0.0439 (17) | 0.0424 (18) | 0.062 (2) | 0.0007 (15) | 0.0053 (14) | −0.0021 (15) |
| C20 | 0.0451 (17) | 0.053 (2) | 0.061 (2) | −0.0043 (16) | 0.0036 (14) | 0.0053 (16) |
| C21 | 0.057 (2) | 0.065 (2) | 0.0516 (18) | −0.0027 (18) | 0.0019 (15) | 0.0040 (17) |
| C22 | 0.069 (2) | 0.054 (2) | 0.055 (2) | −0.0101 (18) | 0.0092 (17) | −0.0098 (17) |
| C23 | 0.075 (3) | 0.063 (3) | 0.075 (3) | 0.014 (2) | −0.010 (2) | 0.003 (2) |
| C24 | 0.072 (3) | 0.071 (3) | 0.081 (3) | −0.019 (2) | −0.011 (2) | 0.002 (2) |
| C25 | 0.074 (2) | 0.0439 (19) | 0.065 (2) | −0.0057 (19) | −0.0016 (17) | 0.0081 (17) |
| C26 | 0.072 (2) | 0.057 (2) | 0.058 (2) | 0.025 (2) | 0.0082 (17) | −0.0017 (18) |
| C27 | 0.0536 (19) | 0.0434 (19) | 0.066 (2) | −0.0041 (15) | −0.0010 (16) | 0.0010 (16) |
| C28 | 0.064 (2) | 0.0377 (18) | 0.062 (2) | 0.0028 (16) | 0.0095 (16) | −0.0004 (16) |
| C29 | 0.078 (3) | 0.066 (3) | 0.070 (2) | 0.029 (2) | 0.002 (2) | −0.006 (2) |
| C30 | 0.075 (3) | 0.067 (3) | 0.075 (2) | 0.012 (2) | −0.003 (2) | 0.011 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C28 | 1.351 (4) | C15—C16 | 1.534 (5) |
| O1—C13 | 1.502 (4) | C15—H15A | 0.970 |
| O2—C28 | 1.216 (5) | C15—H15B | 0.970 |
| O3—C3 | 1.421 (4) | C16—C17 | 1.537 (5) |
| O3—H3A | 0.820 | C16—H16A | 0.970 |
| C1—C2 | 1.536 (5) | C16—H16B | 0.970 |
| C1—C10 | 1.547 (4) | C17—C28 | 1.511 (5) |
| C1—H1A | 0.970 | C17—C22 | 1.520 (5) |
| C1—H1B | 0.970 | C17—C18 | 1.536 (4) |
| C2—C3 | 1.506 (5) | C18—C19 | 1.542 (5) |
| C2—H2A | 0.970 | C18—H18A | 0.980 |
| C2—H2B | 0.970 | C19—C29 | 1.525 (5) |
| C3—C4 | 1.537 (5) | C19—C20 | 1.555 (5) |
| C3—H3B | 0.980 | C19—H19A | 0.980 |
| C4—C23 | 1.533 (5) | C20—C30 | 1.525 (6) |
| C4—C24 | 1.537 (5) | C20—C21 | 1.527 (5) |
| C4—C5 | 1.568 (5) | C20—H20A | 0.980 |
| C5—C6 | 1.532 (4) | C21—C22 | 1.527 (6) |
| C5—C10 | 1.556 (4) | C21—H21A | 0.970 |
| C5—H5A | 0.980 | C21—H21B | 0.970 |
| C6—C7 | 1.528 (5) | C22—H22A | 0.970 |
| C6—H6A | 0.970 | C22—H22B | 0.970 |
| C6—H6B | 0.970 | C23—H23A | 0.960 |
| C7—C8 | 1.545 (5) | C23—H23B | 0.960 |
| C7—H7A | 0.970 | C23—H23C | 0.960 |
| C7—H7B | 0.970 | C24—H24A | 0.960 |
| C8—C9 | 1.559 (4) | C24—H24B | 0.960 |
| C8—C26 | 1.562 (5) | C24—H24C | 0.960 |
| C8—C14 | 1.614 (4) | C25—H25A | 0.960 |
| C9—C11 | 1.541 (4) | C25—H25B | 0.960 |
| C9—C10 | 1.569 (4) | C25—H25C | 0.960 |
| C9—H9A | 0.980 | C26—H26A | 0.960 |
| C10—C25 | 1.558 (5) | C26—H26B | 0.960 |
| C11—C12 | 1.522 (5) | C26—H26C | 0.960 |
| C11—H11A | 0.970 | C27—H27A | 0.960 |
| C11—H11B | 0.970 | C27—H27B | 0.960 |
| C12—C13 | 1.510 (4) | C27—H27C | 0.960 |
| C12—H12A | 0.970 | C29—H29A | 0.960 |
| C12—H12B | 0.970 | C29—H29B | 0.960 |
| C13—C14 | 1.563 (4) | C29—H29C | 0.960 |
| C13—C18 | 1.573 (4) | C30—H30A | 0.960 |
| C14—C27 | 1.549 (5) | C30—H30B | 0.960 |
| C14—C15 | 1.552 (4) | C30—H30C | 0.960 |
| | | | |
| C28—O1—C13 | 109.7 (2) | C14—C15—H15B | 108.6 |
| C3—O3—H3A | 109.5 | H15A—C15—H15B | 107.5 |

| | | | |
|---|---|---|---|
| C2—C1—C10 | 112.7 (3) | C15—C16—C17 | 113.6 (3) |
| C2—C1—H1A | 109.0 | C15—C16—H16A | 108.8 |
| C10—C1—H1A | 109.0 | C17—C16—H16A | 108.8 |
| C2—C1—H1B | 109.0 | C15—C16—H16B | 108.8 |
| C10—C1—H1B | 109.0 | C17—C16—H16B | 108.8 |
| H1A—C1—H1B | 107.8 | H16A—C16—H16B | 107.7 |
| C3—C2—C1 | 112.8 (3) | C28—C17—C22 | 114.3 (3) |
| C3—C2—H2A | 109.0 | C28—C17—C18 | 98.5 (3) |
| C1—C2—H2A | 109.0 | C22—C17—C18 | 112.6 (3) |
| C3—C2—H2B | 109.0 | C28—C17—C16 | 107.9 (3) |
| C1—C2—H2B | 109.0 | C22—C17—C16 | 113.0 (3) |
| H2A—C2—H2B | 107.8 | C18—C17—C16 | 109.5 (3) |
| O3—C3—C2 | 111.3 (3) | C17—C18—C19 | 113.2 (3) |
| O3—C3—C4 | 108.7 (3) | C17—C18—C13 | 99.6 (2) |
| C2—C3—C4 | 113.9 (3) | C19—C18—C13 | 128.1 (3) |
| O3—C3—H3B | 107.6 | C17—C18—H18A | 104.6 |
| C2—C3—H3B | 107.6 | C19—C18—H18A | 104.6 |
| C4—C3—H3B | 107.6 | C13—C18—H18A | 104.6 |
| C23—C4—C24 | 107.6 (3) | C29—C19—C18 | 112.5 (3) |
| C23—C4—C3 | 111.2 (3) | C29—C19—C20 | 110.6 (3) |
| C24—C4—C3 | 107.0 (3) | C18—C19—C20 | 108.1 (3) |
| C23—C4—C5 | 113.5 (3) | C29—C19—H19A | 108.5 |
| C24—C4—C5 | 109.5 (3) | C18—C19—H19A | 108.5 |
| C3—C4—C5 | 107.9 (3) | C20—C19—H19A | 108.5 |
| C6—C5—C10 | 110.2 (3) | C30—C20—C21 | 109.6 (3) |
| C6—C5—C4 | 113.9 (3) | C30—C20—C19 | 112.0 (3) |
| C10—C5—C4 | 118.4 (3) | C21—C20—C19 | 111.5 (3) |
| C6—C5—H5A | 104.2 | C30—C20—H20A | 107.9 |
| C10—C5—H5A | 104.2 | C21—C20—H20A | 107.9 |
| C4—C5—H5A | 104.2 | C19—C20—H20A | 107.9 |
| C7—C6—C5 | 110.7 (3) | C20—C21—C22 | 113.3 (3) |
| C7—C6—H6A | 109.5 | C20—C21—H21A | 108.9 |
| C5—C6—H6A | 109.5 | C22—C21—H21A | 108.9 |
| C7—C6—H6B | 109.5 | C20—C21—H21B | 108.9 |
| C5—C6—H6B | 109.5 | C22—C21—H21B | 108.9 |
| H6A—C6—H6B | 108.1 | H21A—C21—H21B | 107.7 |
| C6—C7—C8 | 114.2 (3) | C17—C22—C21 | 110.0 (3) |
| C6—C7—H7A | 108.7 | C17—C22—H22A | 109.7 |
| C8—C7—H7A | 108.7 | C21—C22—H22A | 109.7 |
| C6—C7—H7B | 108.7 | C17—C22—H22B | 109.7 |
| C8—C7—H7B | 108.7 | C21—C22—H22B | 109.7 |
| H7A—C7—H7B | 107.6 | H22A—C22—H22B | 108.2 |
| C7—C8—C9 | 108.9 (3) | C4—C23—H23A | 109.5 |
| C7—C8—C26 | 106.2 (3) | C4—C23—H23B | 109.5 |
| C9—C8—C26 | 110.9 (3) | H23A—C23—H23B | 109.5 |
| C7—C8—C14 | 109.7 (3) | C4—C23—H23C | 109.5 |
| C9—C8—C14 | 108.8 (2) | H23A—C23—H23C | 109.5 |
| C26—C8—C14 | 112.2 (3) | H23B—C23—H23C | 109.5 |

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|---|---|---|---|
| C11—C9—C8 | 109.3 (3) | C4—C24—H24A | 109.5 |
| C11—C9—C10 | 114.6 (3) | C4—C24—H24B | 109.5 |
| C8—C9—C10 | 117.0 (2) | H24A—C24—H24B | 109.5 |
| C11—C9—H9A | 104.9 | C4—C24—H24C | 109.5 |
| C8—C9—H9A | 104.9 | H24A—C24—H24C | 109.5 |
| C10—C9—H9A | 104.9 | H24B—C24—H24C | 109.5 |
| C1—C10—C5 | 106.7 (3) | C10—C25—H25A | 109.5 |
| C1—C10—C25 | 107.4 (3) | C10—C25—H25B | 109.5 |
| C5—C10—C25 | 114.3 (3) | H25A—C25—H25B | 109.5 |
| C1—C10—C9 | 108.2 (3) | C10—C25—H25C | 109.5 |
| C5—C10—C9 | 107.1 (2) | H25A—C25—H25C | 109.5 |
| C25—C10—C9 | 112.7 (3) | H25B—C25—H25C | 109.5 |
| C12—C11—C9 | 111.4 (3) | C8—C26—H26A | 109.5 |
| C12—C11—H11A | 109.3 | C8—C26—H26B | 109.5 |
| C9—C11—H11A | 109.3 | H26A—C26—H26B | 109.5 |
| C12—C11—H11B | 109.3 | C8—C26—H26C | 109.5 |
| C9—C11—H11B | 109.3 | H26A—C26—H26C | 109.5 |
| H11A—C11—H11B | 108.0 | H26B—C26—H26C | 109.5 |
| C13—C12—C11 | 114.7 (3) | C14—C27—H27A | 109.5 |
| C13—C12—H12A | 108.6 | C14—C27—H27B | 109.5 |
| C11—C12—H12A | 108.6 | H27A—C27—H27B | 109.5 |
| C13—C12—H12B | 108.6 | C14—C27—H27C | 109.5 |
| C11—C12—H12B | 108.6 | H27A—C27—H27C | 109.5 |
| H12A—C12—H12B | 107.6 | H27B—C27—H27C | 109.5 |
| O1—C13—C12 | 104.9 (3) | O2—C28—O1 | 120.0 (3) |
| O1—C13—C14 | 106.9 (2) | O2—C28—C17 | 130.0 (3) |
| C12—C13—C14 | 113.2 (3) | O1—C28—C17 | 110.0 (3) |
| O1—C13—C18 | 97.8 (2) | C19—C29—H29A | 109.5 |
| C12—C13—C18 | 115.0 (3) | C19—C29—H29B | 109.5 |
| C14—C13—C18 | 116.7 (2) | H29A—C29—H29B | 109.5 |
| C27—C14—C15 | 107.5 (3) | C19—C29—H29C | 109.5 |
| C27—C14—C13 | 107.8 (3) | H29A—C29—H29C | 109.5 |
| C15—C14—C13 | 108.3 (3) | H29B—C29—H29C | 109.5 |
| C27—C14—C8 | 111.0 (3) | C20—C30—H30A | 109.5 |
| C15—C14—C8 | 110.5 (3) | C20—C30—H30B | 109.5 |
| C13—C14—C8 | 111.5 (2) | H30A—C30—H30B | 109.5 |
| C16—C15—C14 | 114.9 (3) | C20—C30—H30C | 109.5 |
| C16—C15—H15A | 108.6 | H30A—C30—H30C | 109.5 |
| C14—C15—H15A | 108.6 | H30B—C30—H30C | 109.5 |
| C16—C15—H15B | 108.6 | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3*A*···O2$^{i}$ | 0.82 | 2.24 | 3.059 (4) | 176 |

Symmetry code: (i) −*x*, *y*+1/2, −*z*+2.