# (1S,2R,8R)-2,2-Dichloro-3,7,7,10-tetra-methyltricyclo[6.4.0.0$^{1,3}$]dodec-10-en-9-one

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The title compound, $C_{16}H_{22}Cl_2O$, was synthesized from $\beta$-himachalene, which was isolated from essential oil of the Atlas cedar (*cedrus atlantica*). The asymmetric unit contains two independent molecules, in each of which the six-membered ring shows a half-chair conformation, whereas the seven-membered ring displays a boat conformation. The dihedral angle between the two rings is slightly different in the two molecules [63.22 (13) and 61.81 (14)°].

## Related literature

For the isolation of $\beta$-himachalene, see: Joseph & Dev (1968); Plattier & Teiseire (1974). For the reactivity of this sesqui-terpene, see: Lassaba *et al.* (1998); Chekroun *et al.* (2000); El Jamili *et al.* (2002); Sbai *et al.* (2002); Dakir *et al.* (2004). For its biological activity, see: Daoubi *et al.* (2004).
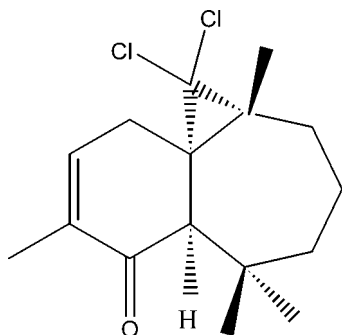


## Experimental

### Crystal data

| | |
|---|---|
| $C_{16}H_{22}Cl_2O$ | $V$ = 1598.7 (3) Å$^3$ |
| $M_r$ = 301.24 | $Z$ = 4 |
| Monoclinic, $P2_1$ | Mo $K\alpha$ radiation |
| $a$ = 6.6680 (7) Å | $\mu$ = 0.40 mm$^{-1}$ |
| $b$ = 18.7760 (16) Å | $T$ = 298 K |
| $c$ = 12.7696 (12) Å | $0.27 \times 0.18 \times 0.12$ mm |
| $\beta$ = 90.430 (3)° | |

### Data collection

| | |
|---|---|
| Bruker X8 APEXII CCD area-detector diffractometer | 5817 independent reflections |
| | 4348 reflections with $I > 2\sigma(I)$ |
| 11524 measured reflections | $R_{int}$ = 0.031 |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.039 | $\Delta\rho_{max}$ = 0.17 e Å$^{-3}$ |
| $wR(F^2)$ = 0.094 | $\Delta\rho_{min}$ = −0.17 e Å$^{-3}$ |
| $S$ = 1.00 | Absolute structure: Flack & Bernardinelli (2000), 1791 Friedel pairs |
| 5817 reflections | |
| 351 parameters | |
| 1 restraint | Flack parameter: 0.01 (5) |
| H-atom parameters constrained | |

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; 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).

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# supporting information

# (1*S*,2*R*,8*R*)-2,2-Dichloro-3,7,7,10-tetramethyltricyclo[6.4.0.0$^{1,3}$]dodec-10-en-9-one

## Ahmed Benharref, Lahcen El Ammari and Moha Berraho

## S1. Comment

The essential oil of the Atlas cedar (*cedrus atlantica*) consists mainly (50%) of a bicyclic hydrocarbon sesquiterpene called *β*-himachalene (Joseph & Dev 1968; Plattier & Teiseire 1974). The reactivity of this sesquiterpene has been studied extensively by our team in order to prepare new products having biological proprieties (Lassaba *et al.*, 1998; Chekroun *et al.*, 2000; El Jamili *et al.*, 2002; Sbai *et al.*, 2002; Dakir *et al.*, 2004). Indeed, these compounds were tested, using the food poisoning technique, for their potential antifungal activity against phytopathogen Botrytis cinerea (Daoubi *et al.*, 2004). Thus the action of one equivalent of dichlorocarbene, generated *in situ* from chloroform and in the presence of sodium hydroxide as base and n-benzyltriethylammonium chloride as catalyst, followed by epoxydation with *m*-chloro-perbenzoic acid (*m*-CPBA) gives a mixture of two diasteroisomers:(1*S*,3*R*,8*S*,9*S*,10*R*)-2,2-dichloro-9,10-epoxy-3,7,7,10-tetramethyltricyclo[6.4.0.0$^{1,3}$]dodecane (*X*, 70%) and (1*S*,3*R*,8*S*,9*R*,10*S*)-2,2-dichloro-9,10-epoxy-3,7,7,10-tetramethyltricyclo[6.4.0.0$^{1,3}$]dodecane (*Y*, 30%) (Sbai *et al.*, 2002). By treatment of the majority isomer (*X*) with hydrochloric acid (see experimental) we obtained the title compound in 70% yield. The structure of (I) was established by $^1$H and $^{13}$C NMR and confirmed by its single crystal X-ray structure. The asymmetric unit contains two crystallographically independent molecules (Fig.1). Each molecule is built up from two fused six and seven-membered rings with the dichlorocyclopropyl ring at positions 1 and 2 in *α*-configuration. The six-membered ring shows a half chair conformation, whereas the seven-membered ring displays a boat conformation. In the first molecule (C1 to C16), the dihedral angle between the rings is 63.22 (13)°. The corresponding value in the second molecule (C17 to C31) is 61.81 (14)°. Owing to the presence of the Cl atoms, the absolute configuration could be fully confirmed to be (1*S*,2*R*,8*R*) for both molecules (Flack & Bernardinelli, 2000).

## S2. Experimental

A solution is prepared by bubbling gaseous hydrochloric acid into 40 ml of chloroform for 1 minute. Then 1 g (3.30 mmol) of (1*S*,3*R*,8*S*,9*S*,10*R*)-2,2-dichloro-9,10-epoxy-3,7,7,10-tetramethyltricyclo[6.4.0.0$^{1,3}$]dodecane (*X*) dissolved in 20 ml of chloroform, is added to this solution and the resulting mixture is stirred for 1 h. After evaporation of the solvent and chromatography of the residue obtained on a column of silica gel, using as eluent hexane-ethyl acetate (95/ 5), the corresponding enol is isolated (yield: 0.6 g, 1.98 mmol, 60%). A solution of the enol (0.5 g, 1.65 mmol) in dichloro-methane (20 ml) was added to a suspension of the complex (CrO$_3$: pyridine) (10 mmol) in another 45 ml of dichloro-methane. The reaction mixture was stirred at room temperature for 1 h, then filtered through an alumina column. After evaporation of the solvent, the title compound, (1*S*,2*R*,8*R*)-2,2-Dichloro-3,7,7,10-tetramethyl-tricyclo-[6.4.0.0$^{1,3}$]dodec-10-en-9-one, is obtained (0.35 g, 70%). Crystals suitable for X-ray analysis were produced by recrystallization from *n*-pentane.

## S3. Refinement

All H atoms were fixed geometrically and treated as riding with C—H = 0.96 Å (methyl),0.97 Å (methylene), 0.98Å (methine) with $U_{iso}(H) = 1.2U_{eq}$ (methylene, methine) or $U_{iso}(H) = 1.5U_{eq}$ (methyl).
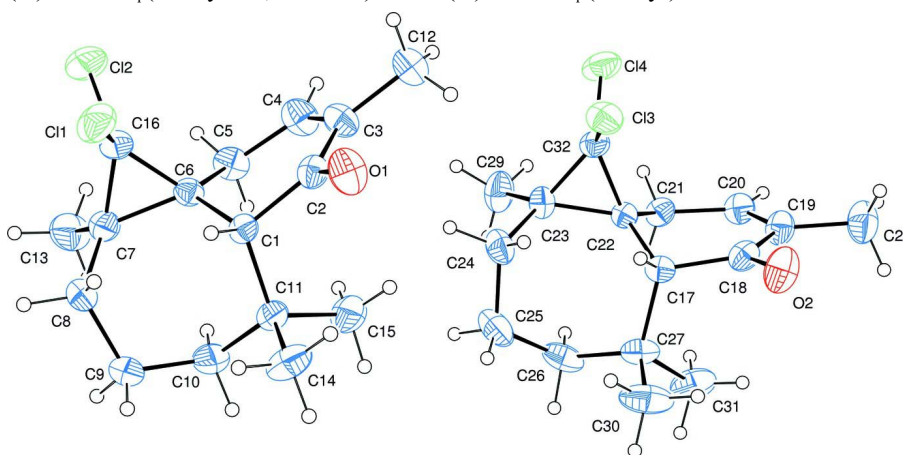


**Figure 1**

Molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

**(1*S*,2*R*,8*R*)-2,2-Dichloro-3,7,7,10- tetramethyltricyclo[6.4.0.0$^{1,3}$]dodec-10-en-9-one**

*Crystal data*

| | |
|---|---|
| $C_{16}H_{22}Cl_2O$ | $F(000) = 640$ |
| $M_r = 301.24$ | $D_x = 1.252$ Mg m$^{-3}$ |
| Monoclinic, $P2_1$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: P 2yb | Cell parameters from 11524 reflections |
| $a = 6.6680$ (7) Å | $\theta = 2.3$–28.2° |
| $b = 18.7760$ (16) Å | $\mu = 0.40$ mm$^{-1}$ |
| $c = 12.7696$ (12) Å | $T = 298$ K |
| $\beta = 90.430$ (3)° | Prism, colourless |
| $V = 1598.7$ (3) Å$^3$ | $0.27 \times 0.18 \times 0.12$ mm |
| $Z = 4$ | |

*Data collection*

| | |
|---|---|
| Bruker X8 APEXII CCD area-detector diffractometer | 4348 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{int} = 0.031$ |
| Graphite monochromator | $\theta_{max} = 28.2°$, $\theta_{min} = 2.7°$ |
| $\varphi$ and $\omega$ scans | $h = -8 \rightarrow 8$ |
| 11524 measured reflections | $k = -16 \rightarrow 24$ |
| 5817 independent reflections | $l = -14 \rightarrow 17$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | 1 restraint |
| Least-squares matrix: full | Primary atom site location: structure-invariant direct methods |
| $R[F^2 > 2\sigma(F^2)] = 0.039$ | |
| $wR(F^2) = 0.094$ | Secondary atom site location: difference Fourier map |
| $S = 1.00$ | |
| 5817 reflections | Hydrogen site location: inferred from neighbouring sites |
| 351 parameters | |

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0451P)^2]$

   where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.17$ e Å$^{-3}$

$\Delta\rho_{min} = -0.17$ e Å$^{-3}$

Absolute structure: Flack & Bernardinelli

   (2000), 1791 Friedel pairs

Absolute structure parameter: 0.01 (5)

*Special details*

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

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C4 | 0.2648 (4) | 0.51700 (18) | 0.1518 (2) | 0.0665 (8) |
| H4 | 0.3713 | 0.5344 | 0.1916 | 0.080* |
| C5 | 0.3032 (4) | 0.50170 (17) | 0.0397 (2) | 0.0566 (7) |
| H5A | 0.3645 | 0.4551 | 0.0335 | 0.068* |
| H5B | 0.3967 | 0.5366 | 0.0125 | 0.068* |
| C6 | 0.1141 (4) | 0.50357 (13) | −0.02434 (19) | 0.0428 (5) |
| C7 | 0.1190 (4) | 0.50653 (15) | −0.1455 (2) | 0.0555 (7) |
| C13 | 0.3194 (5) | 0.5040 (2) | −0.2006 (3) | 0.0842 (11) |
| H13A | 0.3063 | 0.5251 | −0.2688 | 0.126* |
| H13B | 0.3615 | 0.4553 | −0.2077 | 0.126* |
| H13C | 0.4173 | 0.5298 | −0.1603 | 0.126* |
| C29 | 0.7542 (5) | 0.3406 (2) | 0.3751 (3) | 0.0802 (10) |
| H29A | 0.7532 | 0.3825 | 0.3320 | 0.120* |
| H29B | 0.7934 | 0.3003 | 0.3338 | 0.120* |
| H29C | 0.8478 | 0.3469 | 0.4318 | 0.120* |
| C30 | 0.1340 (6) | 0.1455 (2) | 0.4271 (3) | 0.1001 (13) |
| H30A | 0.0623 | 0.1353 | 0.4903 | 0.150* |
| H30B | 0.1376 | 0.1036 | 0.3841 | 0.150* |
| H30C | 0.0677 | 0.1832 | 0.3898 | 0.150* |
| C32 | 0.4841 (4) | 0.36421 (13) | 0.51889 (19) | 0.0483 (6) |
| Cl2 | 0.23340 (15) | 0.64403 (4) | −0.07935 (7) | 0.0867 (3) |
| Cl3 | 0.24108 (11) | 0.40022 (3) | 0.53257 (6) | 0.0636 (2) |
| Cl4 | 0.65593 (14) | 0.42095 (4) | 0.58207 (7) | 0.0779 (2) |
| C1 | −0.0602 (3) | 0.46308 (12) | 0.02462 (19) | 0.0401 (5) |
| H1 | −0.1815 | 0.4787 | −0.0126 | 0.048* |
| C2 | −0.0857 (4) | 0.48562 (13) | 0.1377 (2) | 0.0478 (6) |
| C3 | 0.0909 (4) | 0.50787 (16) | 0.1996 (2) | 0.0577 (7) |
| C8 | −0.0460 (5) | 0.46708 (15) | −0.2027 (2) | 0.0635 (8) |
| H8A | −0.0446 | 0.4806 | −0.2760 | 0.076* |
| H8B | −0.1744 | 0.4812 | −0.1742 | 0.076* |
| C9 | −0.0246 (6) | 0.38591 (16) | −0.1941 (2) | 0.0753 (9) |
| H9A | −0.1549 | 0.3645 | −0.2070 | 0.090* |
| H9B | 0.0644 | 0.3698 | −0.2490 | 0.090* |
| C10 | 0.0552 (5) | 0.35865 (15) | −0.0899 (2) | 0.0681 (8) |

| | | | |
|---|---|---|---|
| H10A | 0.1938 | 0.3738 | −0.0837 | 0.082* |
| H10B | 0.0560 | 0.3070 | −0.0933 | 0.082* |
| C11 | −0.0502 (4) | 0.37973 (13) | 0.0114 (2) | 0.0476 (6) |
| C12 | 0.0569 (6) | 0.52838 (19) | 0.3121 (2) | 0.0791 (10) |
| H12A | 0.1817 | 0.5263 | 0.3499 | 0.119* |
| H12B | −0.0371 | 0.4960 | 0.3430 | 0.119* |
| H12C | 0.0043 | 0.5759 | 0.3151 | 0.119* |
| C14 | −0.2671 (5) | 0.35177 (17) | 0.0103 (3) | 0.0793 (10) |
| H14A | −0.2660 | 0.3008 | 0.0045 | 0.119* |
| H14B | −0.3383 | 0.3718 | −0.0483 | 0.119* |
| H14C | −0.3322 | 0.3653 | 0.0741 | 0.119* |
| C15 | 0.0620 (5) | 0.34352 (16) | 0.1020 (3) | 0.0709 (9) |
| H15A | 0.0584 | 0.2928 | 0.0925 | 0.106* |
| H15B | −0.0008 | 0.3557 | 0.1670 | 0.106* |
| H15C | 0.1989 | 0.3594 | 0.1032 | 0.106* |
| C16 | 0.0640 (4) | 0.57190 (14) | −0.0831 (2) | 0.0545 (7) |
| C17 | 0.3373 (3) | 0.23635 (12) | 0.52663 (18) | 0.0416 (5) |
| H17 | 0.2229 | 0.2643 | 0.5009 | 0.050* |
| C18 | 0.2929 (4) | 0.21847 (13) | 0.6403 (2) | 0.0491 (6) |
| C19 | 0.4595 (4) | 0.21347 (15) | 0.7164 (2) | 0.0566 (7) |
| C20 | 0.6448 (4) | 0.23164 (15) | 0.6869 (2) | 0.0541 (7) |
| H20 | 0.7473 | 0.2279 | 0.7364 | 0.065* |
| C21 | 0.6985 (4) | 0.25743 (14) | 0.5803 (2) | 0.0504 (6) |
| H21A | 0.7596 | 0.2188 | 0.5415 | 0.060* |
| H21B | 0.7965 | 0.2954 | 0.5867 | 0.060* |
| C22 | 0.5185 (4) | 0.28455 (12) | 0.51998 (19) | 0.0394 (5) |
| C23 | 0.5449 (4) | 0.32811 (15) | 0.41890 (19) | 0.0503 (6) |
| C24 | 0.3892 (5) | 0.31700 (17) | 0.3332 (2) | 0.0620 (8) |
| H24A | 0.4055 | 0.3534 | 0.2801 | 0.074* |
| H24B | 0.2564 | 0.3223 | 0.3627 | 0.074* |
| C25 | 0.4058 (6) | 0.2439 (2) | 0.2822 (2) | 0.0831 (10) |
| H25A | 0.5018 | 0.2470 | 0.2256 | 0.100* |
| H25B | 0.2768 | 0.2320 | 0.2513 | 0.100* |
| C26 | 0.4694 (5) | 0.18318 (19) | 0.3547 (3) | 0.0788 (10) |
| H26A | 0.6073 | 0.1919 | 0.3757 | 0.095* |
| H26B | 0.4690 | 0.1398 | 0.3135 | 0.095* |
| C27 | 0.3494 (4) | 0.16840 (15) | 0.4547 (2) | 0.0612 (8) |
| C28 | 0.4103 (5) | 0.1891 (2) | 0.8254 (3) | 0.0821 (10) |
| H28A | 0.5294 | 0.1903 | 0.8681 | 0.123* |
| H28B | 0.3590 | 0.1414 | 0.8230 | 0.123* |
| H28C | 0.3112 | 0.2201 | 0.8549 | 0.123* |
| C31 | 0.4526 (6) | 0.10533 (17) | 0.5119 (3) | 0.0908 (11) |
| H31A | 0.5875 | 0.1184 | 0.5304 | 0.136* |
| H31B | 0.4548 | 0.0646 | 0.4665 | 0.136* |
| H31C | 0.3795 | 0.0940 | 0.5742 | 0.136* |
| O1 | −0.2520 (3) | 0.48319 (13) | 0.17818 (16) | 0.0732 (6) |
| O2 | 0.1224 (3) | 0.20740 (13) | 0.66801 (17) | 0.0771 (6) |
| Cl1 | −0.18216 (13) | 0.60662 (4) | −0.08502 (7) | 0.0734 (2) |

*Atomic displacement parameters (Å²)*

|  | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C4 | 0.0487 (17) | 0.085 (2) | 0.065 (2) | −0.0084 (15) | −0.0153 (14) | −0.0067 (17) |
| C5 | 0.0387 (14) | 0.0655 (18) | 0.0656 (18) | −0.0037 (12) | −0.0004 (12) | −0.0059 (14) |
| C6 | 0.0402 (13) | 0.0434 (13) | 0.0447 (14) | −0.0043 (10) | −0.0006 (11) | −0.0034 (11) |
| C7 | 0.0677 (18) | 0.0501 (15) | 0.0489 (16) | −0.0119 (13) | 0.0092 (13) | −0.0005 (12) |
| C13 | 0.092 (3) | 0.089 (3) | 0.072 (2) | −0.019 (2) | 0.0332 (19) | −0.0136 (19) |
| C29 | 0.072 (2) | 0.100 (3) | 0.070 (2) | −0.0123 (19) | 0.0127 (17) | 0.0220 (19) |
| C30 | 0.077 (2) | 0.087 (3) | 0.137 (3) | −0.019 (2) | −0.014 (2) | −0.052 (3) |
| C32 | 0.0542 (15) | 0.0398 (13) | 0.0506 (16) | 0.0004 (11) | −0.0125 (12) | 0.0009 (11) |
| Cl2 | 0.1082 (7) | 0.0573 (5) | 0.0947 (6) | −0.0378 (5) | 0.0078 (5) | −0.0006 (4) |
| Cl3 | 0.0704 (5) | 0.0544 (4) | 0.0658 (4) | 0.0229 (3) | −0.0103 (3) | −0.0051 (3) |
| Cl4 | 0.0927 (6) | 0.0486 (4) | 0.0918 (6) | −0.0169 (4) | −0.0318 (4) | −0.0002 (4) |
| C1 | 0.0353 (13) | 0.0399 (13) | 0.0449 (14) | 0.0020 (10) | −0.0076 (10) | 0.0039 (11) |
| C2 | 0.0454 (15) | 0.0471 (14) | 0.0511 (16) | 0.0094 (11) | 0.0035 (12) | 0.0059 (12) |
| C3 | 0.0539 (18) | 0.0667 (18) | 0.0522 (16) | 0.0042 (14) | −0.0065 (13) | −0.0101 (14) |
| C8 | 0.089 (2) | 0.0588 (17) | 0.0430 (15) | −0.0148 (15) | −0.0073 (14) | −0.0008 (13) |
| C9 | 0.118 (3) | 0.0553 (18) | 0.0527 (17) | −0.0167 (18) | −0.0016 (17) | −0.0117 (14) |
| C10 | 0.091 (2) | 0.0446 (16) | 0.069 (2) | 0.0051 (15) | −0.0035 (16) | −0.0075 (14) |
| C11 | 0.0488 (15) | 0.0406 (13) | 0.0532 (15) | −0.0014 (11) | −0.0020 (12) | 0.0025 (11) |
| C12 | 0.105 (3) | 0.080 (2) | 0.0529 (18) | −0.003 (2) | −0.0018 (17) | −0.0074 (16) |
| C14 | 0.075 (2) | 0.0495 (17) | 0.114 (3) | −0.0145 (15) | −0.0048 (19) | 0.0074 (17) |
| C15 | 0.081 (2) | 0.0552 (17) | 0.076 (2) | 0.0137 (15) | −0.0104 (17) | 0.0120 (15) |
| C16 | 0.0626 (18) | 0.0459 (14) | 0.0549 (17) | −0.0149 (13) | −0.0024 (13) | −0.0026 (13) |
| C17 | 0.0348 (13) | 0.0408 (13) | 0.0490 (15) | 0.0067 (10) | −0.0050 (10) | −0.0034 (11) |
| C18 | 0.0430 (15) | 0.0421 (14) | 0.0624 (17) | 0.0073 (11) | 0.0050 (12) | 0.0054 (12) |
| C19 | 0.0551 (17) | 0.0587 (17) | 0.0559 (17) | 0.0108 (13) | −0.0002 (13) | 0.0135 (13) |
| C20 | 0.0510 (16) | 0.0544 (15) | 0.0566 (17) | 0.0087 (12) | −0.0141 (13) | 0.0066 (13) |
| C21 | 0.0460 (15) | 0.0466 (14) | 0.0584 (17) | 0.0029 (11) | −0.0054 (12) | 0.0061 (12) |
| C22 | 0.0378 (13) | 0.0395 (12) | 0.0410 (13) | 0.0040 (10) | −0.0019 (10) | −0.0007 (10) |
| C23 | 0.0494 (16) | 0.0582 (16) | 0.0433 (14) | 0.0008 (12) | −0.0001 (12) | 0.0077 (13) |
| C24 | 0.070 (2) | 0.0738 (19) | 0.0416 (15) | 0.0087 (15) | −0.0054 (13) | 0.0035 (15) |
| C25 | 0.102 (3) | 0.098 (3) | 0.0487 (18) | 0.011 (2) | −0.0075 (17) | −0.0172 (18) |
| C26 | 0.088 (2) | 0.073 (2) | 0.076 (2) | 0.0114 (18) | 0.0056 (18) | −0.0394 (18) |
| C27 | 0.0563 (17) | 0.0455 (15) | 0.082 (2) | −0.0012 (12) | −0.0020 (15) | −0.0182 (15) |
| C28 | 0.090 (2) | 0.090 (3) | 0.066 (2) | 0.015 (2) | 0.0108 (18) | 0.0236 (19) |
| C31 | 0.097 (3) | 0.0443 (17) | 0.131 (3) | 0.0114 (18) | 0.003 (2) | −0.010 (2) |
| O1 | 0.0502 (12) | 0.1049 (17) | 0.0647 (13) | 0.0081 (11) | 0.0127 (10) | 0.0001 (12) |
| O2 | 0.0439 (12) | 0.0959 (17) | 0.0917 (16) | 0.0044 (11) | 0.0137 (10) | 0.0280 (13) |
| Cl1 | 0.0823 (6) | 0.0530 (4) | 0.0846 (5) | 0.0118 (4) | −0.0146 (4) | 0.0132 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C4—C3 | 1.326 (4) | C11—C15 | 1.532 (4) |
| C4—C5 | 1.484 (4) | C11—C14 | 1.538 (4) |
| C4—H4 | 0.9300 | C12—H12A | 0.9600 |
| C5—C6 | 1.498 (4) | C12—H12B | 0.9600 |

| | | | |
|---|---|---|---|
| C5—H5A | 0.9700 | C12—H12C | 0.9600 |
| C5—H5B | 0.9700 | C14—H14A | 0.9600 |
| C6—C16 | 1.522 (4) | C14—H14B | 0.9600 |
| C6—C1 | 1.527 (3) | C14—H14C | 0.9600 |
| C6—C7 | 1.549 (4) | C15—H15A | 0.9600 |
| C7—C16 | 1.510 (4) | C15—H15B | 0.9600 |
| C7—C8 | 1.510 (4) | C15—H15C | 0.9600 |
| C7—C13 | 1.516 (4) | C16—Cl1 | 1.766 (3) |
| C13—H13A | 0.9600 | C17—C22 | 1.513 (3) |
| C13—H13B | 0.9600 | C17—C18 | 1.521 (4) |
| C13—H13C | 0.9600 | C17—C27 | 1.574 (3) |
| C29—C23 | 1.526 (4) | C17—H17 | 0.9800 |
| C29—H29A | 0.9600 | C18—O2 | 1.211 (3) |
| C29—H29B | 0.9600 | C18—C19 | 1.473 (4) |
| C29—H29C | 0.9600 | C19—C20 | 1.339 (4) |
| C30—C27 | 1.537 (4) | C19—C28 | 1.505 (4) |
| C30—H30A | 0.9600 | C20—C21 | 1.491 (4) |
| C30—H30B | 0.9600 | C20—H20 | 0.9300 |
| C30—H30C | 0.9600 | C21—C22 | 1.509 (3) |
| C32—C23 | 1.504 (4) | C21—H21A | 0.9700 |
| C32—C22 | 1.513 (3) | C21—H21B | 0.9700 |
| C32—Cl4 | 1.756 (3) | C22—C23 | 1.539 (3) |
| C32—Cl3 | 1.766 (3) | C23—C24 | 1.517 (4) |
| Cl2—C16 | 1.764 (3) | C24—C25 | 1.523 (5) |
| C1—C2 | 1.515 (4) | C24—H24A | 0.9700 |
| C1—C11 | 1.576 (3) | C24—H24B | 0.9700 |
| C1—H1 | 0.9800 | C25—C26 | 1.527 (5) |
| C2—O1 | 1.228 (3) | C25—H25A | 0.9700 |
| C2—C3 | 1.474 (4) | C25—H25B | 0.9700 |
| C3—C12 | 1.506 (4) | C26—C27 | 1.537 (5) |
| C8—C9 | 1.535 (4) | C26—H26A | 0.9700 |
| C8—H8A | 0.9700 | C26—H26B | 0.9700 |
| C8—H8B | 0.9700 | C27—C31 | 1.549 (5) |
| C9—C10 | 1.519 (4) | C28—H28A | 0.9600 |
| C9—H9A | 0.9700 | C28—H28B | 0.9600 |
| C9—H9B | 0.9700 | C28—H28C | 0.9600 |
| C10—C11 | 1.529 (4) | C31—H31A | 0.9600 |
| C10—H10A | 0.9700 | C31—H31B | 0.9600 |
| C10—H10B | 0.9700 | C31—H31C | 0.9600 |
| | | | |
| C3—C4—C5 | 125.3 (3) | C11—C14—H14A | 109.5 |
| C3—C4—H4 | 117.4 | C11—C14—H14B | 109.5 |
| C5—C4—H4 | 117.4 | H14A—C14—H14B | 109.5 |
| C4—C5—C6 | 111.8 (2) | C11—C14—H14C | 109.5 |
| C4—C5—H5A | 109.3 | H14A—C14—H14C | 109.5 |
| C6—C5—H5A | 109.3 | H14B—C14—H14C | 109.5 |
| C4—C5—H5B | 109.3 | C11—C15—H15A | 109.5 |
| C6—C5—H5B | 109.3 | C11—C15—H15B | 109.5 |

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|---|---|---|---|
| H5A—C5—H5B | 107.9 | H15A—C15—H15B | 109.5 |
| C5—C6—C16 | 118.0 (2) | C11—C15—H15C | 109.5 |
| C5—C6—C1 | 113.9 (2) | H15A—C15—H15C | 109.5 |
| C16—C6—C1 | 117.1 (2) | H15B—C15—H15C | 109.5 |
| C5—C6—C7 | 121.5 (2) | C7—C16—C6 | 61.42 (18) |
| C16—C6—C7 | 58.90 (17) | C7—C16—Cl2 | 118.7 (2) |
| C1—C6—C7 | 116.6 (2) | C6—C16—Cl2 | 119.7 (2) |
| C16—C7—C8 | 118.4 (3) | C7—C16—Cl1 | 121.5 (2) |
| C16—C7—C13 | 119.3 (3) | C6—C16—Cl1 | 121.25 (19) |
| C8—C7—C13 | 113.7 (3) | Cl2—C16—Cl1 | 108.17 (15) |
| C16—C7—C6 | 59.67 (17) | C22—C17—C18 | 110.30 (19) |
| C8—C7—C6 | 116.4 (2) | C22—C17—C27 | 114.06 (19) |
| C13—C7—C6 | 119.2 (3) | C18—C17—C27 | 112.9 (2) |
| C7—C13—H13A | 109.5 | C22—C17—H17 | 106.3 |
| C7—C13—H13B | 109.5 | C18—C17—H17 | 106.3 |
| H13A—C13—H13B | 109.5 | C27—C17—H17 | 106.3 |
| C7—C13—H13C | 109.5 | O2—C18—C19 | 120.1 (2) |
| H13A—C13—H13C | 109.5 | O2—C18—C17 | 120.4 (2) |
| H13B—C13—H13C | 109.5 | C19—C18—C17 | 119.5 (2) |
| C23—C29—H29A | 109.5 | C20—C19—C18 | 119.4 (2) |
| C23—C29—H29B | 109.5 | C20—C19—C28 | 123.1 (3) |
| H29A—C29—H29B | 109.5 | C18—C19—C28 | 117.5 (3) |
| C23—C29—H29C | 109.5 | C19—C20—C21 | 124.6 (2) |
| H29A—C29—H29C | 109.5 | C19—C20—H20 | 117.7 |
| H29B—C29—H29C | 109.5 | C21—C20—H20 | 117.7 |
| C27—C30—H30A | 109.5 | C20—C21—C22 | 112.3 (2) |
| C27—C30—H30B | 109.5 | C20—C21—H21A | 109.1 |
| H30A—C30—H30B | 109.5 | C22—C21—H21A | 109.1 |
| C27—C30—H30C | 109.5 | C20—C21—H21B | 109.1 |
| H30A—C30—H30C | 109.5 | C22—C21—H21B | 109.1 |
| H30B—C30—H30C | 109.5 | H21A—C21—H21B | 107.9 |
| C23—C32—C22 | 61.35 (17) | C21—C22—C17 | 113.7 (2) |
| C23—C32—Cl4 | 118.95 (19) | C21—C22—C32 | 117.3 (2) |
| C22—C32—Cl4 | 119.78 (18) | C17—C22—C32 | 118.1 (2) |
| C23—C32—Cl3 | 120.68 (18) | C21—C22—C23 | 120.8 (2) |
| C22—C32—Cl3 | 121.13 (18) | C17—C22—C23 | 117.5 (2) |
| Cl4—C32—Cl3 | 108.56 (14) | C32—C22—C23 | 59.02 (17) |
| C2—C1—C6 | 110.0 (2) | C32—C23—C24 | 119.1 (2) |
| C2—C1—C11 | 112.6 (2) | C32—C23—C29 | 119.7 (3) |
| C6—C1—C11 | 114.7 (2) | C24—C23—C29 | 112.3 (2) |
| C2—C1—H1 | 106.3 | C32—C23—C22 | 59.62 (15) |
| C6—C1—H1 | 106.3 | C24—C23—C22 | 116.7 (2) |
| C11—C1—H1 | 106.3 | C29—C23—C22 | 120.0 (2) |
| O1—C2—C3 | 120.3 (2) | C23—C24—C25 | 112.4 (3) |
| O1—C2—C1 | 119.9 (2) | C23—C24—H24A | 109.1 |
| C3—C2—C1 | 119.7 (2) | C25—C24—H24A | 109.1 |
| C4—C3—C2 | 119.2 (3) | C23—C24—H24B | 109.1 |
| C4—C3—C12 | 123.0 (3) | C25—C24—H24B | 109.1 |

| | | | |
|---|---|---|---|
| C2—C3—C12 | 117.3 (2) | H24A—C24—H24B | 107.9 |
| C7—C8—C9 | 112.7 (3) | C24—C25—C26 | 115.7 (3) |
| C7—C8—H8A | 109.1 | C24—C25—H25A | 108.3 |
| C9—C8—H8A | 109.1 | C26—C25—H25A | 108.3 |
| C7—C8—H8B | 109.1 | C24—C25—H25B | 108.3 |
| C9—C8—H8B | 109.1 | C26—C25—H25B | 108.3 |
| H8A—C8—H8B | 107.8 | H25A—C25—H25B | 107.4 |
| C10—C9—C8 | 115.4 (2) | C25—C26—C27 | 119.7 (3) |
| C10—C9—H9A | 108.4 | C25—C26—H26A | 107.4 |
| C8—C9—H9A | 108.4 | C27—C26—H26A | 107.4 |
| C10—C9—H9B | 108.4 | C25—C26—H26B | 107.4 |
| C8—C9—H9B | 108.4 | C27—C26—H26B | 107.4 |
| H9A—C9—H9B | 107.5 | H26A—C26—H26B | 106.9 |
| C9—C10—C11 | 119.6 (3) | C30—C27—C26 | 110.6 (3) |
| C9—C10—H10A | 107.4 | C30—C27—C31 | 107.7 (3) |
| C11—C10—H10A | 107.4 | C26—C27—C31 | 107.3 (3) |
| C9—C10—H10B | 107.4 | C30—C27—C17 | 108.0 (2) |
| C11—C10—H10B | 107.4 | C26—C27—C17 | 111.6 (2) |
| H10A—C10—H10B | 106.9 | C31—C27—C17 | 111.7 (3) |
| C10—C11—C15 | 107.4 (2) | C19—C28—H28A | 109.5 |
| C10—C11—C14 | 110.0 (2) | C19—C28—H28B | 109.5 |
| C15—C11—C14 | 108.0 (2) | H28A—C28—H28B | 109.5 |
| C10—C11—C1 | 111.6 (2) | C19—C28—H28C | 109.5 |
| C15—C11—C1 | 112.4 (2) | H28A—C28—H28C | 109.5 |
| C14—C11—C1 | 107.4 (2) | H28B—C28—H28C | 109.5 |
| C3—C12—H12A | 109.5 | C27—C31—H31A | 109.5 |
| C3—C12—H12B | 109.5 | C27—C31—H31B | 109.5 |
| H12A—C12—H12B | 109.5 | H31A—C31—H31B | 109.5 |
| C3—C12—H12C | 109.5 | C27—C31—H31C | 109.5 |
| H12A—C12—H12C | 109.5 | H31A—C31—H31C | 109.5 |
| H12B—C12—H12C | 109.5 | H31B—C31—H31C | 109.5 |