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# Stereochemistry of two new polyfunctionalized *gem*-dihalocyclopropanes

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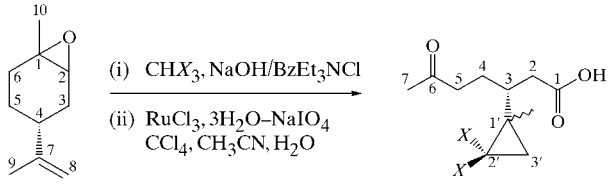
The two new *gem*-dihalogenocyclopropanes (1′*S*,3*R*)-3-(2′,2′-dichloro-1′-methylcyclopropyl)-6-oxoheptanoic acid, $C_{11}H_{16}Cl_2O_3$, (2), and (1′*S*,3*R*)-3-(2′,2′-dibromo-1′-methylcyclopropyl)-6-oxoheptanoic acid, $C_{11}H_{16}Br_2O_3$, (3), are isostructural. Both present two stereogenic centers at C1′ and C3. The absolute configuration was determined by X-ray methods. The cyclopropyl rings are unsymmetrical, the shortest bond being distal with respect to the alkyl-substituted C atom.

## Comment

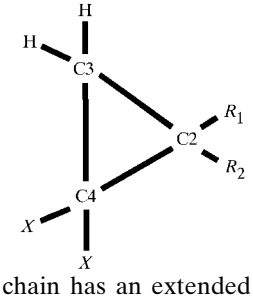
Despite their high ring strain, cyclopropanes are commonly encountered among both naturally occurring and synthetic compounds. In addition, diastereoselectively substituted cyclopropanes have attracted attention as useful precursors of highly strained molecules (Boche *et al.*, 1990; Tanabe *et al.*, 1996) and biologically active pyrethroids (Hirota *et al.*, 1996; Kunzer *et al.*, 1996). Thus, the promise of their usefulness as synthetic intermediates is growing rapidly. We describe here the structure of two new polyfunctionalized dichloro and dibromo cyclopropanes, (2) and (3), which could be valuable synthons for pyrethroid derivatives.




(1)

(2) $X$ = Cl
(3) $X$ = Br

These two compounds were prepared from (*R*)-limonene oxide, (1), by dihalocyclopropanation of the C7=C8 double bond followed by oxidation of the oxirane ring under Sharpless conditions (see *Experimental*). In order to confirm the structure assignments and establish the absolute stereochemistry, single-crystal X-ray studies were carried out on both compounds. The two derivatives crystallize isotypically; only the chloro compound (2) is illustrated in Fig. 1 [a view of the molecule of (3) is given in the supplementary material]. Identical numbering schemes have been employed in both molecules. The absolute configuration (1′*S*,3*R*) has been unambiguously determined by refinement of the Flack (1983) parameter. Examination of the cyclopropyl moieties indicates that the rings are unsymmetrical, with unequal C—C bond lengths (Tables 1 and 3). The C3—C4 bond length in the chloro derivative is 1.479 (2) Å, while the bonds adjacent to the methyl and the polyfunctional substituents are longer [C2—C4 1.504 (3) Å and C2—C3 1.525 (3) Å.] The same trend is observed in the bromo derivative, with identical C—C distances within experimental error. The bond angles within the three-membered ring reflect the difference observed between bond lengths, with the smallest angle at C2 [C3—C2—C4 58.42 (12)°; 59.1(4)° for (3)]. The Cambridge Structural Database (CSD; Allen & Kennard, 1993) has been searched for related dihalogenocyclopropane structures having $CH_2$ and $CR_2$ groups (see *Scheme* below). The search was limited to independent alkyl substituents $R_1$ and $R_2$, and excludes structures with interconnected $R_1$ and $R_2$ for which additional strain might influence the distances within the ring. The geometry of these cyclopropane rings (Table 5) shows the same tendency observed for (2) and (3), with a shortening of the distal bond opposite the alkyl-substituted carbon and a lengthening of the vicinal bonds linking the alkyl and H-substituted C atoms with respect to the mean C—C(ring) length of 1.509 (2) Å (Allen, 1980). These results are in agreement with a previous report (Allen, 1980). The C—Cl bond lengths average 1.762 (2) Å and the C—Br bond lengths average 1.918 (6) Å, in good agreement with related *gem*-dihalogenocyclopropanes, as are the *X*—C—*X* angles of 109.8 (1)° for (2) and 110.1 (3)° for (3).




The 3-oxobutyl chain has an extended configuration, with torsion angles C1—C11—C12—C13 of −180.0 (2)° [−179.0 (6) for (3)] and C11—C12—C13—C14 of 166.2 (2)° [166.3 (6) for (3)] (Tables 1 and 3). The substituents at C1, the carboxyl group and the cyclopropane ring are *anti* with respect to the oxobutyl chain. The orientations of the carboxyl group and the cyclopropane ring are influenced by C—H···O and C—H···Cl (or Br) intramolecular contacts that could be classified as hydrogen bonds. An interaction between an H atom of the cyclopropane C3 atom and O1 of the carboxyl (Tables 2 and 4) results in a twisted conformation of carboxyl

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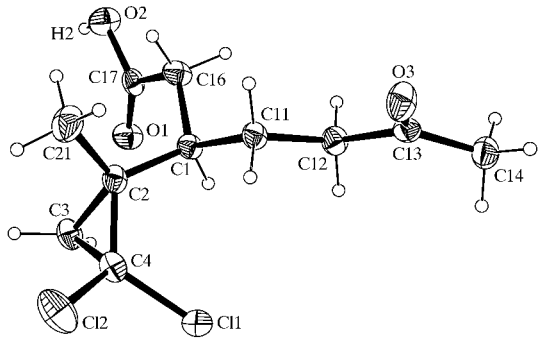


**Figure 1**
*ORTEP*-3 (Farrugia, 1997) view of molecule (2), with displacement ellipsoids at the 50% probability level.

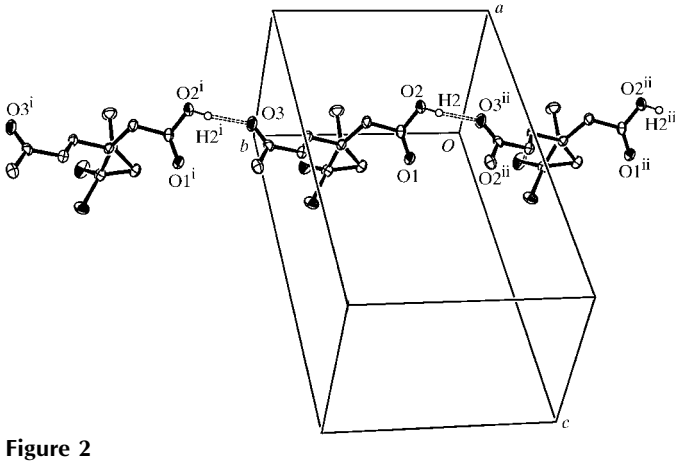


**Figure 2**
Part of the structure of (2), showing the formation of the chain parallel to the *b* axis. For the sake of clarity, H atoms not participating in the hydrogen bonding have been omitted. [Symmetry codes: (i) *x*, *y*−1, *z*; (ii) *x*, *y*+1, *z*.]

group C17/O1/O2 with respect to the C1/C16/C17 plane [dihedral angle 23.1 (2)° for (2) and 24.2° for (3)]. A further interaction occurs between Cl1 and the H atom at C1 (Tables 2 and 4).

Fig. 2 illustrates the packing of (2) in the cell, with extracellular molecules included to show the single-strand hydrogen-bonded catemers. The chain proceeds from the carboxyl group of one molecule to the remote ketone (O3) group of a neighbour (Table 4). Among hydrogen-bonding catemers, the observed prevalence of subtypes describing the relation of adjacent molecules is screw > translation > glide, with the chains often following a cell axis (Brunskill *et al.*, 2001). Here, the components of the chain are related by a translation along *b*. It is noteworthy that there are also short halogen···O1 contacts [3.056(1) Å for the Cl and 3.020 (4) Å for the Br derivative] which connect the hydrogen-bonded catemers.

## Experimental

(*R*)-Limonene oxide, (1), was treated, under phase transfer catalysis conditions, with dichloro(or dibromo)carbene (Tobey & West, 1964) generated *in situ* from the reaction of chloroform (or bromoform) with sodium hydroxide (see *Scheme* in *Comment*). The resulting product was oxidized under Sharpless conditions (Carlson *et al.*, 1981), leading to a diastereoisomeric mixture of 3-(2′,2′-dihalo-1′-methylcyclopropyl)-6-oxoheptanoic acid. Crystals of (2) and (3) were obtained from the corresponding mixture by fractional crystallization from chloroform.

### Compound (2)

*Crystal data*

$C_{11}H_{16}Cl_2O_3$
$M_r$ = 267.14
Orthorhombic, $P2_12_12_1$
$a$ = 7.2419 (5) Å
$b$ = 9.7619 (8) Å
$c$ = 17.7469 (16) Å
$V$ = 1254.61 (18) Å$^3$
$Z$ = 4
$D_x$ = 1.414 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 8000 reflections
$\theta$ = 2.3–26.0°
$\mu$ = 0.51 mm$^{-1}$
$T$ = 180 (2) K
Parallelepiped, colorless
0.42 × 0.40 × 0.13 mm

*Data collection*

Stoe IPDS diffractometer
$\varphi$ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{min}$ = 0.788, $T_{max}$ = 0.913
9901 measured reflections
2418 independent reflections
2207 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.034
$\theta_{max}$ = 26.0°
$h = -8 \rightarrow 8$
$k = -12 \rightarrow 12$
$l = -21 \rightarrow 21$

*Refinement*

Refinement on $F^2$
$R(F)$ = 0.024
$wR(F^2)$ = 0.059
$S$ = 1.04
2418 reflections
148 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0375P)^2 + 0.0454P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.22 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.24 e Å$^{-3}$
Absolute structure: Flack (1983), 2006 Friedel pairs
Flack parameter = 0.00 (5)

**Table 1**
Selected geometric parameters (Å, °) for (2).

| | | | |
|---|---|---|---|
| Cl1—C4 | 1.7668 (17) | C1—C2 | 1.528 (2) |
| Cl2—C4 | 1.7572 (17) | C2—C4 | 1.505 (3) |
| O1—C17 | 1.207 (2) | C2—C21 | 1.511 (2) |
| O2—C17 | 1.325 (2) | C2—C3 | 1.524 (2) |
| O3—C13 | 1.2175 (19) | C3—C4 | 1.478 (2) |
| | | | |
| C4—C2—C21 | 118.20 (15) | C3—C4—Cl2 | 118.73 (12) |
| C4—C2—C3 | 58.42 (11) | C2—C4—Cl2 | 120.84 (13) |
| C21—C2—C3 | 117.78 (14) | C3—C4—Cl1 | 118.80 (12) |
| C4—C2—C1 | 116.65 (14) | C2—C4—Cl1 | 120.02 (12) |
| C21—C2—C1 | 115.61 (16) | Cl2—C4—Cl1 | 109.80 (10) |
| C3—C2—C1 | 118.21 (13) | O1—C17—O2 | 123.05 (14) |
| C4—C3—C2 | 60.11 (12) | O1—C17—C16 | 125.03 (15) |
| C3—C4—C2 | 61.46 (11) | O2—C17—C16 | 111.88 (15) |
| | | | |
| C16—C1—C11—C12 | −81.29 (17) | C2—C1—C16—C17 | −67.65 (18) |
| C2—C1—C11—C12 | 153.51 (15) | C11—C1—C16—C17 | 168.09 (14) |
| C1—C11—C12—C13 | −179.97 (15) | C1—C16—C17—O1 | −24.4 (2) |
| C11—C12—C13—O3 | −14.1 (2) | C1—C16—C17—O2 | 157.88 (14) |
| C11—C12—C13—C14 | 166.17 (16) | | |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H2···O3$^{i}$ | 0.82 | 1.86 | 2.671 (2) | 169 |
| C3—H3$B$···O1 | 0.98 | 2.56 | 3.141 (2) | 118 |
| C1—H1···Cl1 | 0.98 | 2.67 | 3.078 (2) | 105 |

Symmetry code: (i) $x, y-1, z$.

**Table 3**
Selected geometric parameters (Å, °) for (3).

| | | | |
|---|---|---|---|
| Br1—C4 | 1.917 (6) | C1—C2 | 1.537 (8) |
| Br2—C4 | 1.919 (6) | C2—C4 | 1.499 (9) |
| O1—C17 | 1.204 (7) | C2—C3 | 1.517 (8) |
| O2—C17 | 1.321 (7) | C2—C21 | 1.520 (8) |
| O3—C13 | 1.220 (7) | C3—C4 | 1.488 (8) |
| | | | |
| C4—C2—C3 | 59.1 (4) | C3—C4—Br1 | 118.9 (4) |
| C4—C2—C21 | 118.6 (5) | C2—C4—Br1 | 121.1 (4) |
| C3—C2—C21 | 118.7 (5) | C3—C4—Br2 | 116.9 (4) |
| C4—C2—C1 | 116.8 (5) | C2—C4—Br2 | 121.0 (4) |
| C3—C2—C1 | 117.0 (4) | Br1—C4—Br2 | 110.1 (3) |
| C21—C2—C1 | 115.3 (6) | O1—C17—O2 | 122.7 (5) |
| C4—C3—C2 | 59.9 (4) | O1—C17—C16 | 125.1 (5) |
| C3—C4—C2 | 61.0 (4) | O2—C17—C16 | 112.1 (5) |
| | | | |
| C16—C1—C11—C12 | −81.8 (6) | C2—C1—C16—C17 | −67.1 (6) |
| C2—C1—C11—C12 | 151.7 (5) | C11—C1—C16—C17 | 167.2 (4) |
| C1—C11—C12—C13 | 179.0 (6) | C1—C16—C17—O1 | −25.8 (8) |
| C11—C12—C13—O3 | −13.8 (9) | C1—C16—C17—O2 | 157.0 (5) |
| C11—C12—C13—C14 | 166.3 (6) | | |

## Compound (3)

*Crystal data*

$C_{11}H_{16}Br_2O_3$
$M_r$ = 356.06
Orthorhombic, $P2_12_12_1$
$a$ = 7.4006 (5) Å
$b$ = 9.7511 (10) Å
$c$ = 18.0171 (13) Å
$V$ = 1300.19 (19) Å$^3$
$Z$ = 4
$D_x$ = 1.819 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 8000 reflections
$\theta$ = 2.3–26.0°
$\mu$ = 6.23 mm$^{-1}$
$T$ = 180 (2) K
Parallelepiped, colorless
0.30 × 0.28 × 0.18 mm

*Data collection*

Stoe IPDS diffractometer
$\varphi$ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{min}$ = 0.162, $T_{max}$ = 0.348
9311 measured reflections
2376 independent reflections
2070 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.074
$\theta_{max}$ = 26.1°
$h = -8 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -22 \rightarrow 22$

*Refinement*

Refinement on $F^2$
$R(F)$ = 0.037
$wR(F^2)$ = 0.096
$S$ = 1.09
2376 reflections
148 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2 + 0.1506P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.016
$\Delta\rho_{max}$ = 0.51 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.56 e Å$^{-3}$
Absolute structure: Flack (1983), 1972 Friedel pairs
Flack parameter = −0.03 (2)

The crystal of (3) was found to be twinned. However, the two domains could be indexed and the two orientation matrices were used in the integration process (Stoe & Cie, 1996) to produce a set of non-overlapped reflections for each domain. Only the data from the domain with the strongest intensities were retained. As the results were satisfactory, no search for an untwinned crystal was undertaken.

In both compounds, all H atoms were introduced at calculated positions as riding atoms (C—H = 0.97–0.98 Å and OH = 0.82 Å), using *AFIX*37 for $CH_3$ and *AFIX*87 for hydroxyl groups, with a displacement parameter equal to 1.2 (CH and $CH_2$) or 1.5 ($CH_3$ and OH) times that of the parent atom. On the basis of 2006 and 1972 Friedel pairs for compounds (2) and (3), respectively, the final refinement allowed the fraction contribution of the inverted enantiomer to vary (Bernardinelli & Flack, 1985; Flack, 1983), the absolute structure parameter quoted being the refined value of this contribution.

**Table 4**
Hydrogen-bonding geometry (Å, °) for (3).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H2···O3$^{i}$ | 0.82 | 1.87 | 2.676 (5) | 170 |
| C3—H3$B$···O1 | 0.97 | 2.54 | 3.136 (7) | 120 |
| C1—H1···Br1 | 0.98 | 2.76 | 3.194 (6) | 108 |

Symmetry code: (i) $x, y-1, z$.

**Table 5**
Comparison of C—C distances (Å) in some related dihalogenocyclopropanes.

| Compound | $X$ | $R_1$ | $R_2$ | C2—C3 | C2—C4 | C3—C4 |
|---|---|---|---|---|---|---|
| $C_{11}H_{16}Cl_2O_3$[a] | Cl | Me | $C_7H_{11}O_3$ | 1.524 (2) | 1.505 (3) | 1.478 (2) |
| $C_{11}H_{16}Br_2O_3$[a] | Br | Me | $C_7H_{11}O_3$ | 1.517 (8) | 1.499 (9) | 1.488 (8) |
| $C_{12}H_{14}Cl_4O$[b] | Cl | Me | $C_8H_9Cl_2O$ | 1.514 | 1.488 | 1.464 |
| $C_{12}H_{14}Cl_4O$[b] | Cl | Me | $C_8H_9Cl_2O$ | 1.492 | 1.516 | 1.459 |
| $C_{15}H_{10}Cl_4$[c] | Cl | $C_6H_4Cl$ | $C_6H_4Cl$ | 1.484 | 1.472 | 1.473 |
| | Cl | $C_6H_4Cl$ | $C_6H_4Cl$ | 1.517 | 1.543 | 1.546 |
| $C_{15}H_{12}Cl_2$[d] | Cl | Ph | Ph | 1.529 | 1.520 | 1.490 |
| $C_{15}H_{12}Br_2$[d] | Br | Ph | Ph | 1.508 | 1.509 | 1.477 |
| $C_{13}H_{16}Cl_4$[e] | Cl | $CO_2$ | $C_6H_4OEt$ | 1.519 | 1.517 | 1.486 |
| $C_5H_6Cl_2O_2$[f] | Cl | Me | $CO_2$ | 1.523 | 1.523 | 1.481 |
| | Cl | Me | $CO_2$ | 1.520 | 1.520 | 1.483 |
| $C_5H_6Br_2O_2$[f] | Br | Me | $CO_2$ | | | |
| $C_5H_6Cl_2O_2$[f] | Cl | Me | $CH_2CO_2$ | 1.531 | 1.509 | 1.497 |
| $C_{18}H_{14}Cl_4$[g] | Cl | Ph | $C_3H_2PhCl_2$ | 1.520 | 1.505 | 1.472 |
| | Cl | Ph | $C_3H_2PhCl_2$ | 1.525 | 1.508 | 1.472 |
| | Cl | Ph | $C_3H_2PhCl_2$ | 1.527 | 1.535 | 1.469 |
| | Cl | Ph | $C_3H_2PhCl_2$ | 1.516 | 1.533 | 1.477 |
| $C_{18}H_{14}Cl_4$[g] | Cl | Ph | $C_3H_2PhCl_2$ | 1.518 | 1.516 | 1.476 |
| | Cl | Ph | $C_3H_2PhCl_2$ | 1.527 | 1.540 | 1.475 |
| $C_{18}H_{14}Br_4$[g] | Br | Ph | $C_3H_2PhBr_2$ | 1.544 | 1.514 | 1.488 |
| | Br | Ph | $C_3H_2PhBr_2$ | 1.528 | 1.537 | 1.484 |
| $C_{11}H_{10}Cl_2O_2$[h] | Cl | Ph | $CH_2CO_2$ | 1.518 | 1.515 | 1.489 |
| $C_{11}H_{10}Br_2O_2$[h] | Br | Ph | $CH_2CO_2$ | 1.518 | 1.495 | 1.487 |
| $C_{10}H_9BrCl_2$[h] | Cl | Ph | $CH_2Br$ | 1.508 | 1.507 | 1.493 |
| $C_{17}H_{13}Cl_2NO_4$[h] | Cl | Ph | $CO_2CPhNO_2$ | 1.520 | 1.512 | 1.483 |
| $C_5H_7Br_2NO$[i] | Br | $CH_3$ | $CONH_2$ | 1.529 | 1.509 | 1.508 |
| | Br | $CH_3$ | $CONH_2$ | 1.532 | 1.493 | 1.490 |
| $C_{12}H_{13}Cl_2O$[j] | Cl | $CH_3$ | $CHOC_6H_4Me$ | 1.529 | 1.508 | 1.497 |
| | Cl | $CH_3$ | $CHOC_6H_4Me$ | 1.534 | 1.509 | 1.489 |

References: (*a*) this study; (*b*) Zukerman-Schpector *et al.* (1984); (*c*) DeLacy & Kennard (1972); (*d*) Lauher & Ibers (1975); (*e*) Poppleton (1986); (*f*) Romming & Sydnes (1987); (*g*) Lam *et al.* (1997); (*h*) Sydnes *et al.* (1991); (*i*) Baird *et al.* (1999); (*j*) Tanabe *et al.* (1999).

For both compounds, data collection: *IPDS Software* (Stoe & Cie, 1996); cell refinement: *IPDS Software*; data reduction: *XRED* (Stoe & Cie, 1996); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1483). Services for accessing these data are described at the back of the journal.

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