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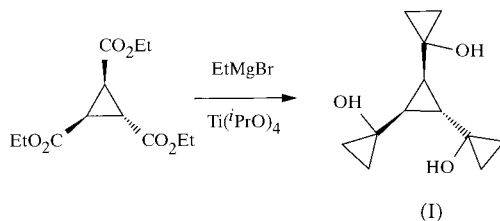
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The central three-membered ring in the title compound, *trans*-1,1',1''-cyclopropane-1,2,3-triyltris(cyclopropanol), C₁₂H₁₈O₃, shows pronounced asymmetry of the bond lengths, which is induced by the different orientations of the substituents. A network of hydrogen bonds links the molecules into sheets.

Comment

trans-1,1',1''-Cyclopropane-1,2,3-triyltris(cyclopropanol), (I), has been synthesized as an intermediate product along the route to tris(cyclopropylidene)cyclopropane in the course of our search for new synthetic approaches to highly strained polyspirocyclopropane derivatives (de Meijere & Kozhushkov, 2000). The compound was obtained in 90% yield from triethyl *trans*-cyclopropanetricarboxylate using the recently developed Kulinkovich reaction (Kulinkovich & de Meijere, 2000) which transforms an alkoxy carbonyl group into cyclopropanol upon treatment with an excess of ethylmagnesium bromide in the presence of Ti(*i*PrO)₄.

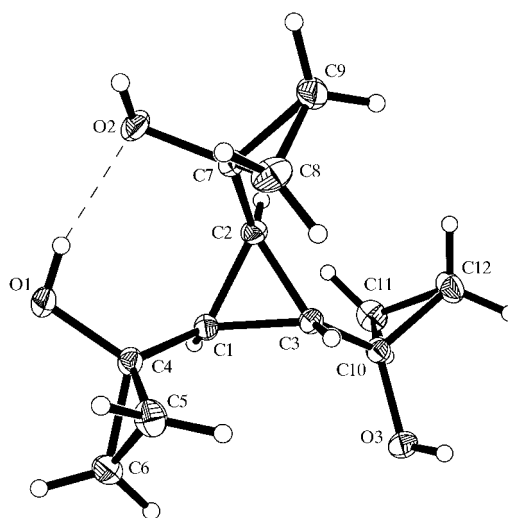


Along with the determination of molecular conformation, the geometry of the symmetrically substituted central cyclopropane (CP) ring was of interest. In molecule (I) we have a relatively rare case of three identical weak π -acceptors connected with three atoms of the central CP ring. In spite of a great number of reported structures of substituted CP, very few examples of such compounds with identical substituents at all three C atoms of CP are available in literature and, to the best of our knowledge, no 1,2,3-tricyclopropylcyclopropane has been described before. The effect of substitution on the

geometry of the CP ring is relatively well understood and described in terms of different models (see, for example, Allen, 1980; Cremer & Kraka, 1985). However, in the case of polysubstituted CP, simple models do not always work. For example, in the systematic study of substituted CP, Jones and Schrupf (1987*a,b,c*) showed that the bond distribution in the three-membered ring depends not solely on the electronic properties of substituents and their individual orientation, but is also strongly affected by stereochemistry of the whole molecule. According to their findings, in the case of a *trans*-conformation of (I), the asymmetry of the ring could be expected. Here we report the results of an X-ray structural study of compound (I) (Fig. 1 and Table 1).

Compound (I) has a *trans*-configuration of substituents in the central three-membered ring with one (at C3) at the opposite side of the ring plane from two others. The bond distribution in the central ring is highly asymmetric with a long bond C1–C2 [1.526 (2) Å] between two *cis*-oriented substituents, an intermediate bond [1.514 (2) Å] and a shorter one [1.504 (2) Å].

The conformations of the substituents are only slightly different. All of them adopt *s-gauche*-configuration around the *exo*-cyclic C–C bonds, when one of the C–C bonds of the terminal ring is in synperiplanar (*sp*) position relative to one of the bonds of the central ring. The conformation is described by torsion angles C2–C3–C10–C11, C3–C1–C4–C5 and C3–C2–C7–C8, which are –20.7, –13.5 and 3.1°, respectively. These similar conformations imply an equal extent of electronic interaction of each of the terminal CP rings with the central one and, indeed, all the CP–CP bonds are identical (mean 1.498 Å). However, it is the mutual orientation of substituents which induces the asymmetry of the central ring. The number of *sp*-oriented C–C bonds of substituents is different for each C–C bond of the central cycle and, therefore, the effect of the substituents on each of them is different. There are two *sp*-orientated bonds for the shortest C2–C3 bond of the central ring, one for the intermediate C1–C3

**Figure 1**

The molecular structure of (I) with displacement ellipsoids at the 50% probability level.

bond and none for the longest C1–C2 one. This particular conformation of the *cis*-substituents is fixed by an intramolecular hydrogen bond O1–H···O2 (Table 2).

The hydrogen bonds determine the packing of molecules in the crystal as well. Intermolecular hydrogen bonds O3–H···O1 and O2–H···O3 link molecules together in loose sheets perpendicular to the (100) direction (Fig. 2). These

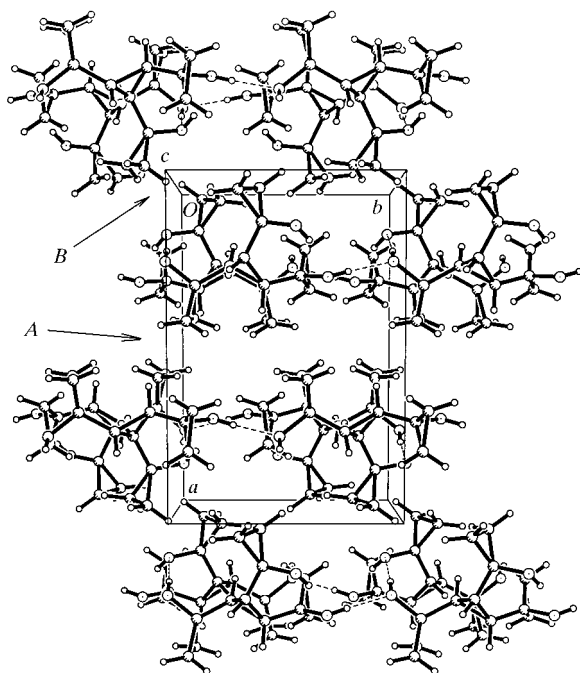


Figure 2
Packing diagram viewed along the *c* axis.

sheets form two types of interface: in the gap marked *A* in Fig. 2, CP rings C10–C12 of the molecules of adjacent layers form contacting surfaces and in the gap of type *B*, CP rings C4–C6 look toward each other. There are no strong intermolecular contacts between the sheets; however, in gap *B* there are contacts [C6–H61···O1 ($2-x, 1-y, -z$); C···O 3.475 (2) Å] which are within the range of typical C–H···O contacts formed by CH groups of three-membered rings (Allen *et al.*, 1996). The shortest interlayer contacts in gap *B* are H2···H112 ($1-x, -\frac{1}{2}+y, \frac{1}{2}-z$) 2.56 (2) Å.

Experimental

Crystals of (I) (de Meijere & Kozhushkov, 2000) were obtained by slow evaporation of a solution in diethyl ether.

Crystal data

C₁₂H₁₈O₃
M_r = 210.26
 Monoclinic, *P*2₁/*c*
a = 12.6464 (3) Å
b = 8.4615 (2) Å
c = 10.4323 (2) Å
 β = 96.578 (1)°
V = 1108.99 (4) Å³
Z = 4

D_x = 1.259 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 511 reflections
 θ = 2.5–29.1°
 μ = 0.09 mm⁻¹
T = 120 (2) K
 Prism, colourless
 0.62 × 0.14 × 0.14 mm

Data collection

Bruker SMART CCD 1 K diffractometer
 ω scans
 10828 measured reflections
 3064 independent reflections
 2555 reflections with $I > 2\sigma(I)$

*R*_{int} = 0.033
 θ_{\max} = 30.3°
h = -17 → 16
k = -11 → 11
l = -14 → 14

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.042
wR(*F*²) = 0.103
S = 1.10
 3064 reflections
 208 parameters
 All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0396P)^2 + 0.4841P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.32 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.23 \text{ e \AA}^{-3}$

Table 1

Selected bond lengths (Å).

O1–C4	1.428 (1)	C4–C6	1.500 (2)
O2–C7	1.424 (1)	C4–C5	1.506 (2)
O3–C10	1.419 (1)	C5–C6	1.524 (2)
C1–C4	1.500 (2)	C7–C9	1.500 (2)
C1–C3	1.514 (2)	C7–C8	1.503 (2)
C1–C2	1.526 (2)	C8–C9	1.522 (2)
C2–C7	1.499 (2)	C10–C11	1.499 (2)
C2–C3	1.504 (2)	C10–C12	1.509 (2)
C3–C10	1.497 (2)	C11–C12	1.522 (2)

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
O1–H1O···O2	0.82 (2)	1.93 (2)	2.729 (1)	166 (2)
O2–H2O···O3 ⁱ	0.86 (2)	1.88 (2)	2.744 (1)	176 (2)
O3–H3O···O1 ⁱⁱ	0.89 (2)	1.83 (2)	2.714 (1)	174 (2)
C6–H61···O1 ⁱⁱⁱ	0.948 (16)	2.584 (16)	3.475 (2)	157 (1)

Symmetry codes: (i) *x, y - 1, z*; (ii) $x, \frac{1}{2} - y, \frac{1}{2} + z$; (iii) $2 - x, 1 - y, -z$.

The range of refined C–H distances is 0.82 (2)–0.99 (2) Å.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SHELXTL* (Bruker, 1997); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD1150). Services for accessing these data are described at the back of the journal.

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