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Key indicators

Single-crystal X-ray study
T = 294 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.040
wR factor = 0.110
Data-to-parameter ratio = 13.0For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.4,7-Dichlorotricyclo[3.1.1.0^{3,6}]heptane-2-carboxylic acid

The structure of the carboxylic acid derivative of the new substituted open-cage system, 4,7-dichlorotricyclo[3.1.1.0^{3,6}]heptane-2-carboxylic acid, $\text{C}_8\text{H}_8\text{Cl}_2\text{O}_2$, shows that the carboxyl C atom has close contacts with C atoms of the cage system. Hydrogen bonds from the carboxylic acid functional groups of adjoining molecules form centrosymmetric dimers.

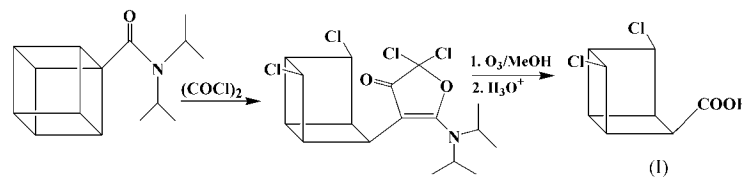
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Comment

There is much current interest in the use of tetrahedrally substituted cubanes in the areas of combinatorial chemistry (Carrell *et al.*, 1994), materials (Chen *et al.*, 1997), and explosives (Eaton *et al.*, 1993; Lukin *et al.*, 1996; Bashir-Hashemi, Iyer *et al.*, 1995; Bashir-Hashemi & Doyle, 1996). In addition, there has been a recent report of another substituted cage system, tricycloheptane (norcubane) (Bashir-Hashemi *et al.*, 1997), obtained from the reaction of 1,4-disubstituted amidocubanes with oxalyl chloride (Bashir-Hashemi, 1993; Bashir-Hashemi, Li *et al.*, 1995).



One of these derivatives, the title compound, 4,7-dichlorotricyclo[3.1.1.0^{3,6}]heptane-2-carboxylic acid, (I) (Bashir-Hashemi *et al.*, 1997), was obtained from the reaction of 1,4-disubstituted amidocubanes with oxalyl chloride giving 4-(4,7-dichlorotricyclo[3.1.1.0^{3,6}]hept-2-yl)-5-[bis(methylethyl)amino]-2,2-dichloro-2-hydrofuran-3-one. The dichloro-2-hydrofuran-3-one was converted to a carboxy group by ozonolysis, followed by acid hydrolysis of the resulting ester. Norcubanes are related to cubanes through the loss of one C–H corner, leaving an open rather than a closed cage system, which should be amenable to further substitution reactions. Compound (I) shows the same substitution pattern as previously observed in these derivatives (Bashir-Hashemi *et al.*, 1997). Similarly, the distances from the carboxyl C atom to opposite corners of the cage (C4 and C7) are 3.136 and 3.132 Å; these are shorter than the non-bonded van der Waals contact distance, which usually ranges from 3.20 to 3.40 Å. This close proximity of both C4 and C7 to C could lead to the construction of desirable molecules, such as azacubanes, by reconstructing the cube corner through a condensation reaction. Compound (I) forms centrosymmetrically linked dimers through hydrogen-bonding interactions of the carboxylic acid groups of adjoining molecules.

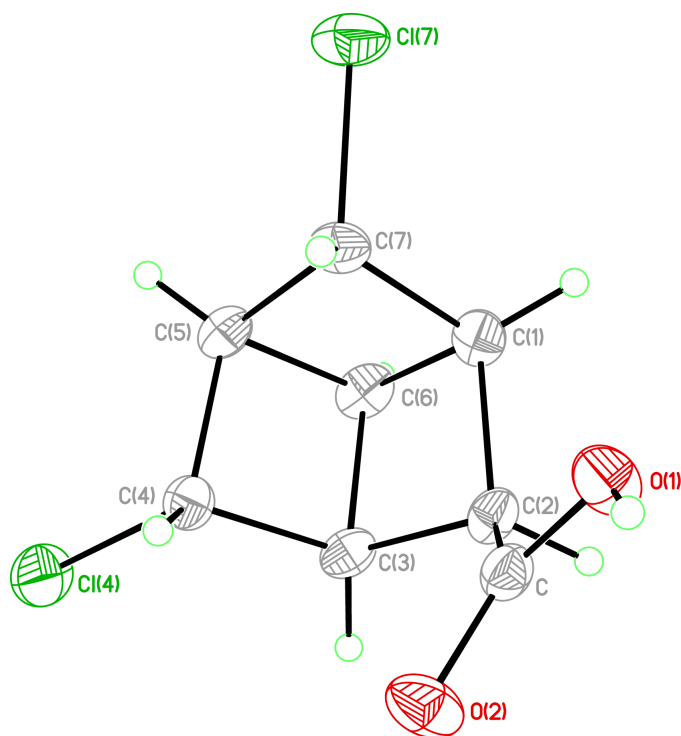


Figure 1
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 20% probability level. H atoms are represented by circles of arbitrary size.

Experimental

Crystals of (I) were obtained by slow evaporation of a methanolic solution of the title compound (m.p. 417–418 K; yield 41%).

Crystal data

$C_8H_8Cl_2O_2$
 $M_r = 207.04$
Monoclinic, $C2/c$
 $a = 21.497(7) \text{ \AA}$
 $b = 9.036(3) \text{ \AA}$
 $c = 10.084(3) \text{ \AA}$
 $\beta = 114.673(6)^\circ$
 $V = 1779.9(10) \text{ \AA}^3$
 $Z = 8$

$D_x = 1.545 \text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 786 reflections
 $\theta = 2.3\text{--}28.1^\circ$
 $\mu = 0.68 \text{ mm}^{-1}$
 $T = 294(2) \text{ K}$
Irregular plate, colourless
 $0.42 \times 0.40 \times 0.10 \text{ mm}$

Data collection

Bruker SMART 1K CCD area-detector diffractometer
 φ and ω scans
Absorption correction: by integration (Wuensch & Prewett, 1965)
 $T_{\min} = 0.740$, $T_{\max} = 0.935$
5852 measured reflections

1822 independent reflections
1512 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$
 $\theta_{\text{max}} = 26.4^\circ$
 $h = -24 \rightarrow 26$
 $k = -11 \rightarrow 6$
 $l = -12 \rightarrow 9$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.110$
 $S = 1.04$
1822 reflections
140 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0521P)^2 + 1.6743P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.053$
 $\Delta\rho_{\text{max}} = 0.39 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.32 \text{ e \AA}^{-3}$

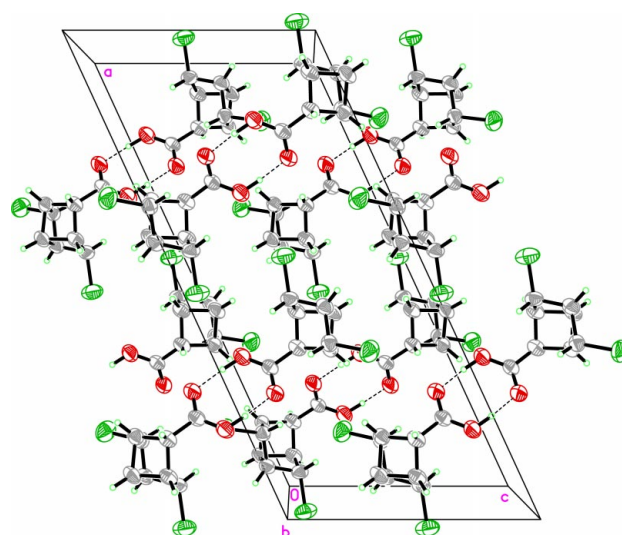


Figure 2
The molecular packing of (I), viewed along the b axis.

Table 1
Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O1-H1O \cdots O2^i$	0.74 (4)	1.90 (4)	2.636 (3)	173 (4)

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

All H atoms were initially located in a difference Fourier map and were refined isotropically.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SHELXTL* (Bruker, 2001); 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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