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Methyl 4,6-dichlorochroman-2-carboxylate

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Key indicators

Single-crystal X-ray study
T = 283 K
Mean σ(C–C) = 0.002 Å
R factor = 0.033
wR factor = 0.091
Data-to-parameter ratio = 16.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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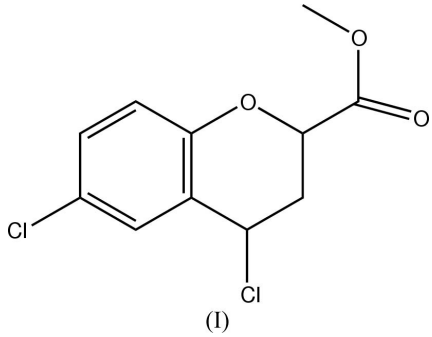
In the title compound, $C_{11}H_{10}Cl_2O_3$, the six-membered heterocyclic ring adopts a half-chair conformation. An intermolecular C—H···O hydrogen bond and π–π stacking are observed in the crystal structure.

Comment

Chroman derivatives exhibit a wide spectrum of biological activity, including antiviral, anticancer and antibiotic properties. The title compound, (I), may be used as a new precursor for obtaining bioactive molecules. In this paper, we present the X-ray crystallographic analysis of (I).



(I)

As shown in Fig. 1, the six-membered heterocyclic ring adopts a half-chair conformation. The puckering parameters (Cremer & Pople, 1975) corresponding to the sequence O1—C4—C5—C7—C8—C9 are Q = 0.470 (2) Å, φ_2 = 88.7 (2)° and θ_2 = 127.81 (2)°. The bond lengths and angles in the molecule are normal.

An intermolecular C—H···O hydrogen bond exists in the crystal structure (Table 1 and Fig. 2), leading to dimerization.

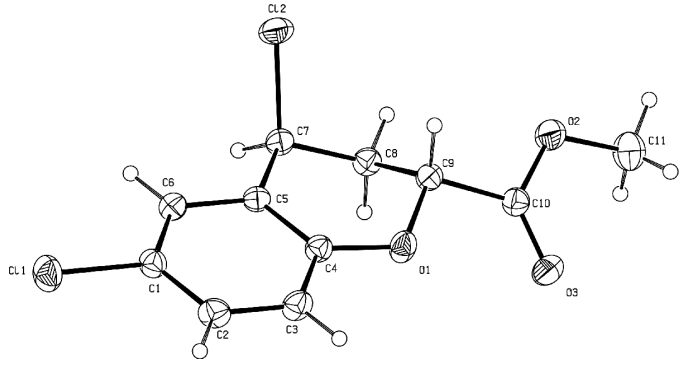


Figure 1
A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size.

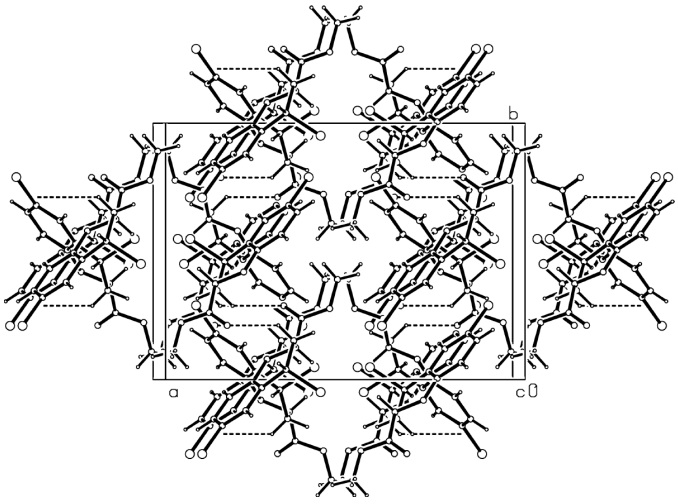


Figure 2
The packing of (I), viewed along the *b* axis. Hydrogen bonds are indicated by dashed lines.

As a result, a ten-membered ring is formed, the topological motif of which corresponds to the first level graph-set descriptor $R_2^2(10)$ (Bernstein *et al.*, 1995). The dihedral angle between the two benzene rings of adjacent molecules is 0.02 (2)° and the distance between the ring centroids is 3.892 (1) Å (Fig. 3). This suggests the existence of a π–π interaction.

Experimental

To a solution of 6-chloro-2-hydroxychromen-4-one (2 mmol) in dichloromethane (10 ml) was added a solution of thionyl chloride (2.4 mmol) in dichloromethane (5 ml). The resulting solution was stirred for 4 h at 298 K. The mixture was then purified by column chromatography on silica gel, with dichloromethane–hexane (7:3 *v*/*v*) as eluent, to afford compound (I) (yield 55%, m.p. 446 K). Spectroscopic analysis: ^{1}H NMR ($CDCl_3$, 400 MHz) 7.28 (*s*, 1H, C5-H), 7.21 (*d*, 1H, C5-H), 6.94 (*d*, 1H, C8-H), 5.20 (*t*, 1H, C4-H), 5.00 (*t*, 1H, C2-H), 3.26 (*s*, 3H, CH_3), 2.44–2.60 (*m*, 2H, CH_2); MS (EI 70 eV) *m*/*z* (%): 264/260 (10/32), 229/225 (5/13), 201 (28), 131/129 (32/28), 102 (100), 74 (90). Crystals suitable for single-crystal X-ray diffraction were grown from acetone at 277 K.

Crystal data

$C_{11}H_{10}Cl_2O_3$	D_x = 1.556 Mg m^{-3}
M_r = 261.09	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 4252 reflections
a = 19.7026 (15) Å	θ = 2.5–27.9°
b = 11.2427 (8) Å	μ = 0.57 mm^{-1}
c = 12.2043 (9) Å	T = 283 (2) K
β = 124.450 (1)°	Block, colourless
V = 2229.3 (3) Å^3	0.60 × 0.30 × 0.20 mm
Z = 8	

Data collection

Bruker SMART 4K CCD area-detector diffractometer	2187 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.019
Absorption correction: none	θ_{max} = 27.0°
6401 measured reflections	$h = -17 \rightarrow 24$
2415 independent reflections	$k = -14 \rightarrow 13$
	$l = -15 \rightarrow 14$

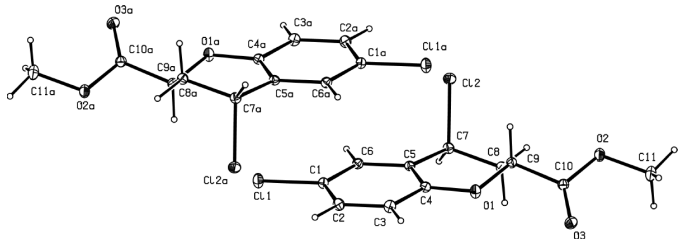


Figure 3
Intermolecular π–π stacking between benzene rings. Atoms labelled with the suffix *a* are at symmetry position ($\frac{1}{2} - x$, $-\frac{1}{2} - y$, $1 - z$).

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2 + 1.1147P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.033	$(\Delta/\sigma)_{max}$ = 0.004
$wR(F^2)$ = 0.092	$\Delta\rho_{max}$ = 0.27 e Å^{-3}
S = 1.04	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
2415 reflections	Extinction correction: *SHELXL97* (Sheldrick, 1997)
147 parameters	Extinction coefficient: 0.0017 (4)
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8A···O3^{i}	0.97	2.57	3.3173 (19)	134

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

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry, with C—H distances of 0.98 Å and U_{iso}(H) = 1.5U_{eq}(C), but each group was allowed to rotate freely about its C—C bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.95–1.00 Å and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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