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Key indicators

Single-crystal X-ray study
T = 295 K
Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
R factor = 0.055
wR factor = 0.157
Data-to-parameter ratio = 13.7

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

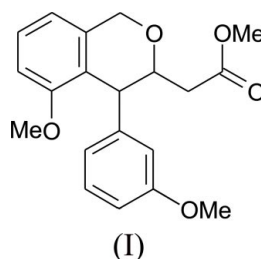
Methyl [5-methoxy-4-(3-methoxyphenyl)-isochroman-3-yl]acetate

In the title compound, $\text{C}_{20}\text{H}_{22}\text{O}_5$, the heterocyclic ring has a half-boat conformation. The methoxycarbonylmethyl substituent is in an equatorial position. The substituents in positions 3 and 4 are in a *trans* configuration.

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Comment

The chemistry of benzo[*c*]pyran derivatives has been recently investigated due to their wide range of biological properties (Moore, 1977; Moore & Czerniak, 1981). The title compound, (I), was prepared during investigations on new precursors to this type of antibiotic (Epsztajn *et al.*, 2001). The present report is part of a continuing structural study of isochroman derivatives (Palusiak *et al.*, 2002).



The molecule of (I) contains two condensed rings, benzene and heterocyclic, with atom O2 in position 2 of the latter. There is a methoxycarbonylmethyl group in position 3, a 3-methoxyphenyl substituent in position 4 and a methoxy group in position 5. The heterocyclic ring can be described by puckering parameters (Cremer & Pople, 1975) corresponding to the sequence O2–C3–C4–C10–C9–C1: $Q = 0.506 (3) \text{ \AA}$, $\varphi_2 = -3.8 (5)^\circ$ and $q_2 = 58.3 (3)^\circ$. The asymmetry parameter (Nardelli, 1983) $\Delta_s(\text{O2})$ is $0.020 (2)$. Ring A has a half-boat conformation (Fig. 1), with the O atom at the apex. The planar methoxycarbonylmethyl moiety is attached equatorially. The substituents in position 3 and 4 are in a *trans* configuration. The torsion angles describing these orientations, C30–C3–C4–C10, C41–C4–C10–C9 and C30–C3–C4–C41, are presented in Table 1. The benzene rings (B and C) are almost planar and form a dihedral angle of $86.1 (1)^\circ$, similar to the dihedral angle of $87.3 (1)^\circ$ between ring C and the condensed moiety (A/B). The angle between the mean plane of the methoxycarbonylmethyl moiety and the mean plane of ring B is $81.7 (1)^\circ$. The methoxy groups attached to the benzene rings are found near the planes of these rings. All bond lengths and angles in (I) are in expected ranges and need no further discussion.

Experimental

The synthesis of (I) has been described elsewhere (Epsztajn *et al.*, 2001). Crystals were obtained by slow evaporation from a methanol solution at room temperature.

Crystal data

$C_{20}H_{22}O_5$	$Z = 2$
$M_r = 342.38$	$D_x = 1.294 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Cu $K\alpha$ radiation
$a = 8.057 (1) \text{ \AA}$	Cell parameters from 25 reflections
$b = 11.145 (2) \text{ \AA}$	$\theta = 22.9\text{--}28.4^\circ$
$c = 11.408 (1) \text{ \AA}$	$\mu = 0.76 \text{ mm}^{-1}$
$\alpha = 68.918 (7)^\circ$	$T = 295 (2) \text{ K}$
$\beta = 71.859 (8)^\circ$	Cut block, colourless
$\gamma = 70.345 (9)^\circ$	$0.32 \times 0.25 \times 0.15 \text{ mm}$
$V = 878.8 (2) \text{ \AA}^3$	

Data collection

Rigaku AFC-5S diffractometer	$R_{\text{int}} = 0.082$
ω scans	$\theta_{\text{max}} = 72.5^\circ$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$h = -9 \rightarrow 9$
$T_{\text{min}} = 0.793$, $T_{\text{max}} = 0.895$	$k = -13 \rightarrow 11$
3500 measured reflections	$l = -14 \rightarrow 13$
3319 independent reflections	3 standard reflections
1732 reflections with $I > 2\sigma(I)$	every 150 reflections
	intensity decay: $<2\%$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0865P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.055$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.157$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 0.87$	$\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$
3319 reflections	$\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$
242 parameters	Extinction correction: <i>SHELXL97</i>
H-atoms parameters constrained	Extinction coefficient: 0.0128 (15)

Table 1

Selected geometric parameters (\AA , $^\circ$).

O2—C3	1.416 (3)	O310—C310	1.449 (3)
O2—C1	1.422 (3)	O43—C43	1.382 (3)
O5—C5	1.366 (3)	O43—C430	1.426 (3)
O5—C50	1.423 (3)	O311—C31	1.202 (3)
O310—C31	1.329 (3)		
C3—O2—C1	111.2 (2)	O2—C3—C30	107.4 (2)
C5—O5—C50	118.0 (2)	O2—C3—C4	113.1 (2)
C31—O310—C310	116.9 (2)	O311—C31—O310	122.9 (3)
C43—O43—C430	117.3 (2)	O311—C31—C30	124.1 (3)
C42—C43—O43	124.4 (3)	O310—C31—C30	113.0 (3)
C10—C4—C3—C30	146.9 (2)	C41—C4—C10—C9	−118.4 (3)
C41—C4—C3—C30	−88.1 (3)		

All H atoms were constrained with a riding model.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1989); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Mol-

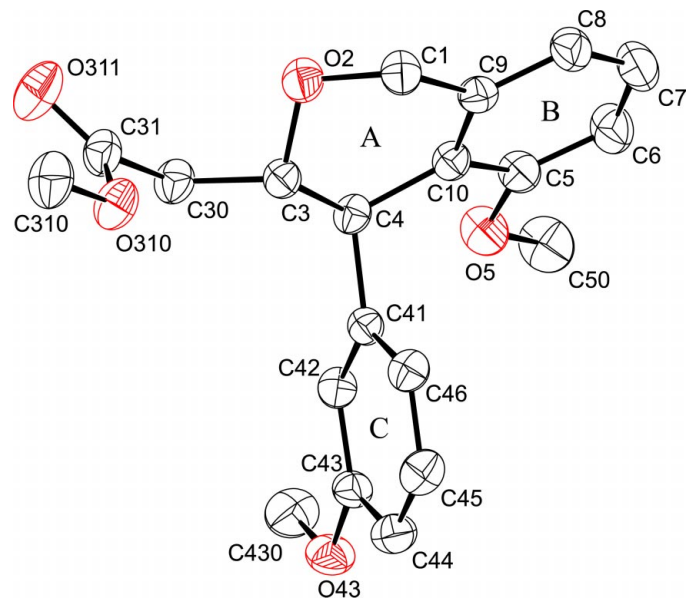


Figure 1

ORTEP drawing of (I), with the atom-labelling scheme. Displacements ellipsoids are drawn at the 40% probability level.

ecular Structure Corporation, 1989); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1998); software used to prepare material for publication: *PARST97* (Nardelli, 1996).

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