Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Pentafluorophenyl 3-chloro-3-phenylpropanoate

Bun Chan, Eng W. Tan and Allan G. Blackman*

Department of Chemistry, University of Otago, PO Box 56, Dunedin, New Zealand

Correspondence e-mail: blackman@alkali.otago.ac.nz

Key indicators

Single-crystal X-ray study
T = 163 K
Mean σ(C–C) = 0.003 Å
R factor = 0.033
wR factor = 0.092
Data-to-parameter ratio = 13.8

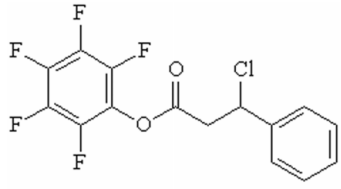
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The structural analysis of the title compound, $C_{15}H_8ClF_5O_2$, confirms the position of the Cl atom at C-3.

Received 21 June 2001
Accepted 5 July 2001
Online 13 July 2001

Comment

The title compound, (I), was investigated as part of a study into an unusual 1,2-chlorine migration that occurs in 2-chlorohydrocinnamate derivatives (Tan *et al.*, 2001). Compound (I) is only the second example of a crystallographically characterized pentafluorophenyl ester, the first being the related compound *N*-*tert*-butyloxycarbonylphenylalanine pentafluorophenyl ester, (II) (Czugler *et al.*, 1976).



(I)

The structure confirms the identity of (I) (Fig. 1) and shows that the Cl atom has migrated from C-2 in the reactant to C-3 in the product. In contrast to (II), where the phenyl and pentafluorophenyl rings are essentially coplanar, the angle between the planes in (I) defined by the phenyl [maximum deviation from its mean plane is 0.0027 (19) Å] and pentafluorophenyl [maximum deviation 0.0008 (15) Å] rings is 53.3 (1)°. The molecules pack as dimers in a 'head-to-tail' fashion, such that there are significant π–π interactions between the phenyl and pentafluorophenyl rings within each dimer, the distances between the ring centroids being 3.826 (3) Å. There is also a short intermolecular distance of 2.368 (2) Å between O2 and the H2*A*^{i} atom of an adjacent molecule [symmetry code: (i) $1-x, y-\frac{1}{2}, \frac{3}{2}-z$].

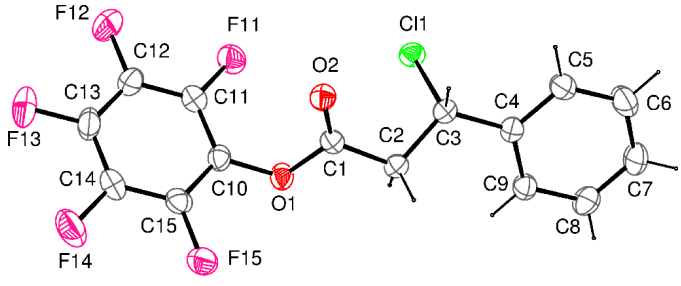


Figure 1
The structure of (I) with displacement ellipsoids drawn at the 50% probability level (Farrugia, 1999).

Experimental

A mixture of pentafluorophenyl (2*R**,3*S**)-3-bromo-2-chloro-3-phenylpropanoate (3.0 g, 8.0 mmol) and tri-*n*-butyltin hydride (4.1 g, 13.8 mmol) in benzene (5 ml) was stirred at room temperature with irradiation from a 160 W mercury gas discharge lamp for 2 h. The reaction mixture was treated with small amounts of iodine until the brown colour of iodine persisted. Aqueous KF (1 *M*, 10 ml) was added to the mixture and the resulting mixture stirred for 2 h. The precipitate formed was removed by filtration and the mixture evaporated under reduced pressure. The residue was purified by column chromatography on silica gel (Sorbsil, particle size 32–63 mm) eluting with CH_2Cl_2. The crude product was recrystallized from hexane to give compound (I) as white crystals, in 53% yield.

Crystal data

$C_{15}H_8ClF_5O_2$	D_x = 1.646 Mg m^{-3}
M_r = 350.66	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 2268 reflections
a = 15.208 (5) Å	θ = 2.9–26.4°
b = 8.131 (5) Å	μ = 0.33 mm^{-1}
c = 12.302 (5) Å	T = 163 (2) K
β = 111.554 (5)°	Rectangular, colourless
V = 1414.8 (11) Å^3	0.68 × 0.32 × 0.29 mm
Z = 4	

Data collection

CCD area-detector diffractometer	R_{int} = 0.032
φ and ω scans	θ_{max} = 26.4°
10557 measured reflections	$h = -18 \rightarrow 18$
2880 independent reflections	$k = -10 \rightarrow 3$
2268 reflections with $I > 2\sigma(I)$	$l = -14 \rightarrow 15$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0563P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.033	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.092	$\Delta\rho_{max}$ = 0.27 e Å^{-3}
S = 1.06	$\Delta\rho_{min}$ = −0.31 e Å^{-3}
2880 reflections	Extinction correction: *SHELXL*97
209 parameters	Extinction coefficient: 0.0040 (12)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.3884 (18)	C3—C4	1.508 (2)
C1—O2	1.1992 (17)	C3—Cl1	1.8463 (16)
C1—C2	1.500 (2)	C10—O1	1.3917 (19)
C2—C3	1.522 (2)		
O2—C1—O1	121.81 (14)	C1—O1—C10	115.41 (11)
O2—C1—C2	127.20 (14)	C2—C3—Cl1	107.64 (10)
O1—C1—C2	110.99 (12)	C4—C3—C2	116.72 (13)
C1—C2—C3	111.00 (12)	C4—C3—Cl1	109.17 (10)

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1996) and *SADABS* (Sheldrick, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *WinGX* (Farrugia, 1999); software used to prepare material for publication: *WinGX*.

We thank Professor W. T. Robinson and Dr Jan Wikaira (University of Canterbury) for data collection. This work is supported by a University of Otago Division of Sciences Research Grant.

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