Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-(3,4-Dichlorobenzylidene)chroman-4-one

Kaalin Gopaul,[a] Neil Anthony Koorbanally,[a]* Mahidansha M. Shaikh,[a] Hong Su[b] and Deresh Ramjugernath[c]

[a]School of Chemistry and Physics, University of KwaZulu-Natal, Private Bag X54001, Durban 4000, South Africa, [b]Chemistry Department, University of Cape Town, Rondebosch, 7701, South Africa, and [c]School of Chemical Engineering, University of KwaZulu-Natal, Durban 4041, South Africa
Correspondence e-mail: Koorbanally@ukzn.ac.za

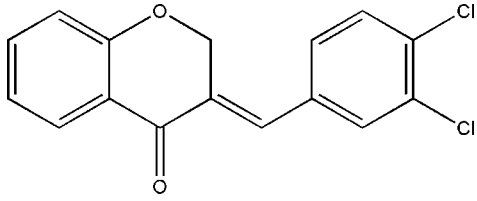
Received 17 September 2012; accepted 25 September 2012

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.034; wR factor = 0.089; data-to-parameter ratio = 18.0.

The distinctive feature of the structure of the title compound, $C_{16}H_{10}Cl_2O_2$, is the formation of a zigzag chain along [100] *via* Cl···Cl interactions [3.591 (1) and 3.631 (1) Å]. The chromanone moiety is fused with the benzene ring and adopts a half-chair conformation. The dihedral angle between the benzene ring of the chromanone moiety and the dichlorobenzene plane is 56.14 (8)°.

Related literature

For background to homoisoflavonoids, see: Kirkiacharian *et al.* (1984). For a related structure, see: Gopaul *et al.* (2012).



Experimental

Crystal data

$C_{16}H_{10}Cl_2O_2$
M_r = 305.14
Monoclinic, $P2_1/c$
a = 3.9224 (3) Å
b = 11.5175 (10) Å
c = 28.957 (3) Å
β = 92.270 (2)°
V = 1307.12 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.49 mm^{-1}
T = 173 K
0.16 × 0.12 × 0.11 mm

Data collection

Bruker Kappa Duo APEXII Diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.925, T_{max} = 0.948
15291 measured reflections
3258 independent reflections
2611 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.089
S = 1.03
3258 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97*.

We thank the University of KwaZulu-Natal and the South Africa Research Chairs initiative of the Department of Science and Technology for financial support and the National Research Foundation of South Africa for a bursary for KG.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5252).

References

Bruker (2006). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Gopaul, K., Shaikh, M., Ramjugernath, D., Koorbanally, N. A. & Omondi, B. (2012). *Acta Cryst.* E**68**, o1006.
Kirkiacharian, B. S., Gomis, M., Tongo, H. G., Mahuteau, J. & Brion, J. D. (1984). *Org. Magn. Reson.* **22**, 106–108.
Sheldrick, G. M. (1997). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2012). E**68**, o3062 [doi:10.1107/S1600536812040561]

3-(3,4-Dichlorobenzylidene)chroman-4-one

Kaalin Gopaul, Neil Anthony Koorbanally, Mahidansha M. Shaikh, Hong Su and Deresh Ramjugernath

S1. Comment

The title compound, 3-(3,4-dichlorobenzylidene)chroman-4-one ($C_{16}H_{10}Cl_2O_2$), belongs to a class of compounds called homoisoflavonoids, which are C-16, α,β unsaturated carbonyl compounds containing two aromatic rings. They are a group of naturally occurring molecules that are structurally related to isoflavonoids but differ by containing one more carbon atom (Kirkiacharian *et al.*, 1984; Gopaul *et al.*, 2012).

A view of the the title compound is shown in Fig. 1. The chromanone moiety is fused with the benzene ring and adopts a half chair conformation. The dihedral angle between the benzene ring of the chromanone moiety and the dichlorobenzene plane is 56.14 (8)°. The inversion-related molecules are linked into zigzag chains *via* Cl···Cl interactions between Cl2 at (*x*, *y*, *z*) and Cl2 at (1 - *x*, 1 - *y*, 1 - *z*) and Cl2 at (-*x*, 1 - *y*, 1 - *z*) with distances of 3.591 (1) Å and 3.631 (1) Å respectively. Molecules related by translation along the *a* axis stack *via* double $\pi\cdots\pi$ interactions of the aromatic rings, with a centroid distance equal to the length of *a* axis. This feature is illustrated in Fig. 2.

S2. Experimental

A mixture of chroman-4-one (1 g, 6.749 mmol), 3,4-dichlorobenzaldehyde (1.417 g, 8.099 mmol) and 10–15 drops of piperidine was heated at 80°C for 18 hrs. The reaction mixture was monitored for completion by thin layer chromatography. Upon completion, the reaction mixture was cooled, diluted with water and neutralized using 10% HCl. The reaction mixture was extracted with ethyl acetate (3 × 30 ml). The ethyl acetate layers were combined, washed with brine (20 ml), water (2 × 10 ml) and dried over anhydrous magnesium sulfate. The solvent was reduced and the compound purified by column chromatography using silica gel (Merck 9385, 40–63 μm particle size) with a mobile phase of 2% ethyl acetate in hexane to yield the title compound with a m.p. of 165–167 °C.

^{1}H NMR: δ (p.p.m.): 5.27 (2*H*, d, *J* = 1.88 Hz, H-2), 6.96 (1*H*, d, *J* = 8.28 Hz, H-8), 7.07 (1*H*, td, *J* = 7.52, 0.68 Hz, H-6), 7.12 (1*H*, dd, *J* = 8.28, 1.96 Hz, H-6′), 7.38 (1*H*, d, *J* = 1.92 Hz, H-2′), 7.49 (1*H*, ddd, *J* = 8.72, 7.48, 1.72 Hz, H-7), 7.50 (1*H*, d, *J* = 8.40 Hz, H-5′), 7.72 (1*H*, s, H-9), 8.00 (1*H*, dd, *J* = 7.88, 1.64 Hz, H-5)

^{13}C NMR: δ (p.p.m.): 67.27 (C-2), 118.00 (C-8), 121.79 (C-4a), 122.16 (C-6), 128.00 (C-5), 128.94 (C-6′), 130.79 (C-5′), 131.41 (C-2′), 132.44 (C-3), 133.15 (C-1′), 133.69 (C-3′), 134.28 (C-4′), 134.55 (C-9), 136.19 (C-7), 161.13 (C-8a), 181.71(C-4)

S3. Refinement

All hydrogen atoms were placed in geometrically idealized positions and constrainted to ride on their parent atoms, with aromatic C—H = 0.95 Å and methylene C—H = 0.99 Å; U_{iso}(H) = 1.2U_{eq}(C).

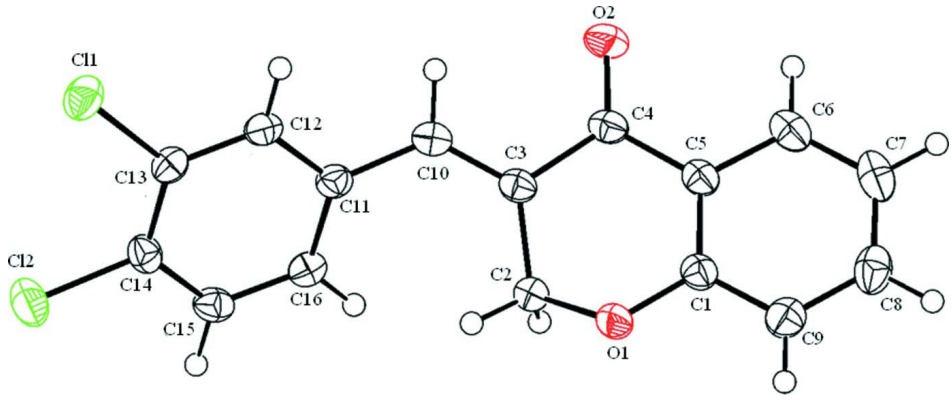


Figure 1

Molecular structure of the title compound, showing the atom labelling scheme and with displacement ellipsoids drawn at the 50% probability level.

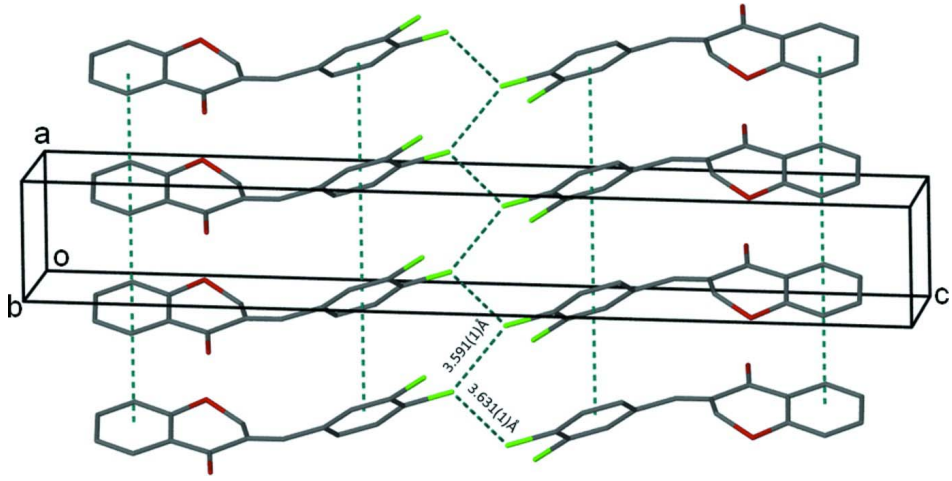


Figure 2

Partial packing diagram showing the Cl···Cl interactions and $\pi\cdots\pi$ stacking as dashed lines. H atoms have been omitted.

3-(3,4-Dichlorobenzylidene)chroman-4-one

Crystal data

$C_{16}H_{10}Cl_2O_2$
$M_r = 305.14$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 3.9224\ (3)$ Å
$b = 11.5175\ (10)$ Å
$c = 28.957\ (3)$ Å
$\beta = 92.270\ (2)°$
$V = 1307.12\ (19)$ Å^3
$Z = 4$
$F(000) = 624$
$D_x = 1.551$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 15291 reflections
$\theta = 1.9–28.3°$
$\mu = 0.49$ mm^{-1}
$T = 173$ K
Block, colourless
0.16 × 0.12 × 0.11 mm

Data collection

Bruker Kappa Duo APEXII Diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
0.5° φ scans and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)

T_{min} = 0.925, T_{max} = 0.948
15291 measured reflections
3258 independent reflections
2611 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

θ_{max} = 28.3°, θ_{min} = 1.9°
h = −5→5
k = −15→15
l = −38→38

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.089
S = 1.03
3258 reflections
181 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0416P)^2 + 0.370P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.42734 (11)	0.87066 (4)	0.446666 (14)	0.03559 (12)
Cl2	0.24634 (12)	0.61056 (4)	0.471571 (14)	0.04001 (13)
O1	0.1216 (3)	0.60873 (9)	0.19421 (4)	0.0295 (3)
O2	−0.4016 (3)	0.91508 (10)	0.20528 (4)	0.0370 (3)
C1	0.0018 (4)	0.66516 (13)	0.15546 (5)	0.0257 (3)
C2	−0.0674 (4)	0.62604 (13)	0.23556 (5)	0.0264 (3)
H2A	0.0689	0.5962	0.2625	0.032*
H2B	−0.2815	0.5806	0.2330	0.032*
C3	−0.1530 (4)	0.75127 (12)	0.24381 (5)	0.0246 (3)
C4	−0.2625 (4)	0.82037 (13)	0.20229 (6)	0.0263 (3)
C5	−0.1875 (4)	0.76778 (13)	0.15740 (5)	0.0253 (3)
C6	−0.2890 (4)	0.82258 (14)	0.11584 (6)	0.0316 (4)
H6	−0.4186	0.8922	0.1166	0.038*
C7	−0.2037 (5)	0.77697 (16)	0.07401 (6)	0.0374 (4)
H7	−0.2737	0.8148	0.0461	0.045*
C8	−0.0143 (5)	0.67520 (16)	0.07285 (6)	0.0365 (4)
H8	0.0453	0.6437	0.0439	0.044*
C9	0.0882 (4)	0.61927 (14)	0.11313 (6)	0.0315 (3)
H9	0.2174	0.5496	0.1120	0.038*
C10	−0.1366 (4)	0.80455 (13)	0.28501 (5)	0.0269 (3)

H10	−0.1923	0.8848	0.2848	0.032*
C11	−0.0434 (4)	0.75431 (13)	0.33026 (5)	0.0253 (3)
C12	0.1296 (4)	0.82387 (13)	0.36316 (5)	0.0258 (3)
H12	0.1865	0.9015	0.3555	0.031*
C13	0.2187 (4)	0.78142 (13)	0.40650 (5)	0.0256 (3)
C14	0.1346 (4)	0.66795 (14)	0.41804 (5)	0.0263 (3)
C15	−0.0449 (4)	0.59937 (13)	0.38621 (6)	0.0275 (3)
H15	−0.1075	0.5226	0.3944	0.033*
C16	−0.1336 (4)	0.64150 (13)	0.34272 (5)	0.0261 (3)
H16	−0.2565	0.5936	0.3212	0.031*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0386 (2)	0.0369 (2)	0.0310 (2)	−0.00723 (17)	−0.00110 (17)	−0.00681 (16)
Cl2	0.0470 (3)	0.0436 (3)	0.0291 (2)	−0.00285 (19)	−0.00218 (18)	0.00968 (17)
O1	0.0354 (6)	0.0268 (6)	0.0265 (6)	0.0085 (4)	0.0024 (5)	0.0026 (4)
O2	0.0447 (7)	0.0258 (6)	0.0400 (7)	0.0113 (5)	−0.0050 (6)	0.0018 (5)
C1	0.0265 (7)	0.0234 (7)	0.0269 (8)	−0.0051 (6)	−0.0014 (6)	0.0021 (6)
C2	0.0314 (8)	0.0232 (7)	0.0245 (7)	0.0025 (6)	0.0013 (6)	0.0011 (6)
C3	0.0226 (7)	0.0227 (7)	0.0283 (8)	0.0016 (5)	−0.0007 (6)	0.0020 (6)
C4	0.0247 (7)	0.0217 (7)	0.0321 (8)	0.0006 (6)	−0.0032 (6)	0.0020 (6)
C5	0.0236 (7)	0.0240 (7)	0.0279 (8)	−0.0047 (6)	−0.0030 (6)	0.0034 (6)
C6	0.0283 (8)	0.0314 (8)	0.0348 (9)	−0.0041 (6)	−0.0046 (7)	0.0082 (7)
C7	0.0363 (9)	0.0467 (10)	0.0286 (9)	−0.0110 (8)	−0.0049 (7)	0.0108 (7)
C8	0.0387 (9)	0.0440 (10)	0.0271 (8)	−0.0118 (8)	0.0044 (7)	−0.0016 (7)
C9	0.0332 (8)	0.0298 (8)	0.0317 (9)	−0.0066 (7)	0.0052 (7)	−0.0013 (6)
C10	0.0262 (7)	0.0224 (7)	0.0322 (8)	0.0013 (6)	0.0014 (6)	0.0013 (6)
C11	0.0264 (7)	0.0232 (7)	0.0265 (8)	0.0030 (6)	0.0044 (6)	−0.0008 (6)
C12	0.0282 (8)	0.0207 (7)	0.0287 (8)	0.0000 (6)	0.0052 (6)	−0.0021 (6)
C13	0.0242 (7)	0.0266 (7)	0.0261 (8)	−0.0008 (6)	0.0030 (6)	−0.0038 (6)
C14	0.0258 (7)	0.0299 (8)	0.0233 (7)	0.0020 (6)	0.0036 (6)	0.0039 (6)
C15	0.0274 (8)	0.0224 (7)	0.0330 (8)	−0.0016 (6)	0.0058 (6)	0.0021 (6)
C16	0.0257 (7)	0.0239 (7)	0.0287 (8)	−0.0010 (6)	0.0020 (6)	−0.0032 (6)

Geometric parameters (Å, º)

Cl1—C13	1.7330 (15)	C7—C8	1.389 (3)
Cl2—C14	1.7256 (15)	C7—H7	0.9500
O1—C1	1.3641 (18)	C8—C9	1.378 (2)
O1—C2	1.4470 (19)	C8—H8	0.9500
O2—C4	1.2243 (19)	C9—H9	0.9500
C1—C9	1.389 (2)	C10—C11	1.465 (2)
C1—C5	1.398 (2)	C10—H10	0.9500
C2—C3	1.502 (2)	C11—C16	1.398 (2)
C2—H2A	0.9900	C11—C12	1.400 (2)
C2—H2B	0.9900	C12—C13	1.379 (2)
C3—C10	1.341 (2)	C12—H12	0.9500

C3—C4	1.491 (2)	C13—C14	1.392 (2)
C4—C5	1.474 (2)	C14—C15	1.385 (2)
C5—C6	1.402 (2)	C15—C16	1.381 (2)
C6—C7	1.374 (3)	C15—H15	0.9500
C6—H6	0.9500	C16—H16	0.9500

C1—O1—C2	116.35 (12)	C9—C8—H8	119.6
O1—C1—C9	117.15 (14)	C7—C8—H8	119.6
O1—C1—C5	122.42 (14)	C8—C9—C1	119.71 (16)
C9—C1—C5	120.37 (15)	C8—C9—H9	120.1
O1—C2—C3	112.89 (12)	C1—C9—H9	120.1
O1—C2—H2A	109.0	C3—C10—C11	128.05 (14)
C3—C2—H2A	109.0	C3—C10—H10	116.0
O1—C2—H2B	109.0	C11—C10—H10	116.0
C3—C2—H2B	109.0	C16—C11—C12	118.51 (14)
H2A—C2—H2B	107.8	C16—C11—C10	122.76 (14)
C10—C3—C4	118.39 (13)	C12—C11—C10	118.66 (13)
C10—C3—C2	125.29 (14)	C13—C12—C11	121.01 (14)
C4—C3—C2	116.32 (13)	C13—C12—H12	119.5
O2—C4—C5	122.25 (14)	C11—C12—H12	119.5
O2—C4—C3	122.24 (15)	C12—C13—C14	119.84 (14)
C5—C4—C3	115.51 (13)	C12—C13—Cl1	119.77 (12)
C1—C5—C6	118.63 (15)	C14—C13—Cl1	120.38 (12)
C1—C5—C4	120.50 (13)	C15—C14—C13	119.66 (14)
C6—C5—C4	120.79 (14)	C15—C14—Cl2	118.93 (12)
C7—C6—C5	120.90 (16)	C13—C14—Cl2	121.41 (12)
C7—C6—H6	119.5	C16—C15—C14	120.64 (14)
C5—C6—H6	119.5	C16—C15—H15	119.7
C6—C7—C8	119.56 (16)	C14—C15—H15	119.7
C6—C7—H7	120.2	C15—C16—C11	120.31 (14)
C8—C7—H7	120.2	C15—C16—H16	119.8
C9—C8—C7	120.82 (16)	C11—C16—H16	119.8

C2—O1—C1—C9	−156.47 (14)	C7—C8—C9—C1	−0.1 (2)
C2—O1—C1—C5	26.3 (2)	O1—C1—C9—C8	−177.45 (14)
C1—O1—C2—C3	−46.21 (17)	C5—C1—C9—C8	−0.2 (2)
O1—C2—C3—C10	−138.89 (16)	C4—C3—C10—C11	178.67 (15)
O1—C2—C3—C4	40.74 (18)	C2—C3—C10—C11	−1.7 (3)
C10—C3—C4—O2	−15.1 (2)	C3—C10—C11—C16	−36.4 (2)
C2—C3—C4—O2	165.28 (15)	C3—C10—C11—C12	146.59 (17)
C10—C3—C4—C5	164.10 (14)	C16—C11—C12—C13	1.9 (2)
C2—C3—C4—C5	−15.56 (19)	C10—C11—C12—C13	179.04 (14)
O1—C1—C5—C6	177.47 (13)	C11—C12—C13—C14	−0.4 (2)
C9—C1—C5—C6	0.3 (2)	C11—C12—C13—Cl1	−179.05 (12)
O1—C1—C5—C4	0.7 (2)	C12—C13—C14—C15	−1.4 (2)
C9—C1—C5—C4	−176.41 (14)	Cl1—C13—C14—C15	177.26 (12)
O2—C4—C5—C1	173.56 (15)	C12—C13—C14—Cl2	179.08 (12)
C3—C4—C5—C1	−5.6 (2)	Cl1—C13—C14—Cl2	−2.23 (19)

O2—C4—C5—C6	−3.1 (2)	C13—C14—C15—C16	1.6 (2)
C3—C4—C5—C6	177.72 (14)	Cl2—C14—C15—C16	−178.85 (12)
C1—C5—C6—C7	−0.3 (2)	C14—C15—C16—C11	−0.1 (2)
C4—C5—C6—C7	176.48 (15)	C12—C11—C16—C15	−1.7 (2)
C5—C6—C7—C8	0.0 (2)	C10—C11—C16—C15	−178.69 (15)
C6—C7—C8—C9	0.2 (3)		