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4-[4-(Benzyloxy)phenyl]-1,1,1-trifluoro-4-hydroxybut-3-en-2-one

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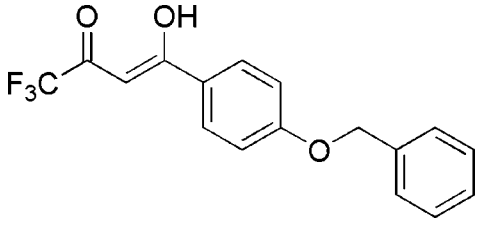
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.006 Å; disorder in main residue; R factor = 0.057; wR factor = 0.152; data-to-parameter ratio = 7.9.

The molecule of the title compound, $C_{17}H_{13}F_3O_3$, exists in the enol form and displays a strong intramolecular O—H···O hydrogen bond. The trifluoromethyl group is disordered over two orientations in an approximate 2:1 ratio.

Related literature

For general background, see: Rowley *et al.* (1996); Shavaleev *et al.* (2003). For related structures, see: Gilli *et al.* (2004); Wang *et al.* (2006).



Experimental

Crystal data

$C_{17}H_{13}F_3O_3$
M_r = 322.27
Orthorhombic, $P2_12_12_1$
a = 7.6226 (11) Å
b = 11.0708 (15) Å
c = 17.748 (2) Å
V = 1497.7 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 294 (2) K
0.20 × 0.10 × 0.10 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.976, T_{max} = 0.983
6737 measured reflections
1881 independent reflections
1110 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.152
S = 1.06
1881 reflections
239 parameters
66 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H1···O3	1.26 (6)	1.29 (6)	2.496 (5)	158 (5)

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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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2164).

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supplementary materials

Acta Cryst. (2007). E**63**, o2979 [**doi:10.1107/S1600536807024737**]

4-[4-(Benzyloxy)phenyl]-1,1,1-trifluoro-4-hydroxybut-3-en-2-one

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Comment

1,3-Diketones are important intermediates not only as a key building block for the synthesis of core heterocycles in medicinal chemistry (Rowley *et al.*, 1996) but also as an invaluable chelating ligand for various lanthanide and transition metals in material chemistry (Shavaleev *et al.*, 2003). The molecular structure of the title compound, (I), exists in the enolized form and displays a strong intramolecular hydrogen bond (Fig. 1 and Table 2). The geometric data (Table 1) are in agreement with reported literature values (Gilli *et al.*, 2004; Wang *et al.*, 2006). The CF_3 group is disordered.

Experimental

The mixture of 1-[4-(benzyloxy)phenyl]ethanone (9.10 g, 0.04 mol), ethyl trifluoracetate (10 ml, 0.084 mol), CH_3ONa (4.32 g, 0.08 mol) and benzene (150 ml) was stirred at 323 K for 8 h. The reaction mixture was cooled to room temperature, acidified with dilute hydrochloric acid and then stirred until all solids dissolved. The benzene layer was separated, washed with a saturated $NaHCO_3$ solution, dried over anhydrous Mg_2SO_4 and the solvent was removed by evaporation. The residual oil solidified on standing and the solid was recrystallized from ethanol to give the title compound (yield 10.10 g, 78.4%; m.p. 367 K). Single crystals suitable for X-ray diffraction were grown by slow evaporation of an ethanol solution at room temperature. Spectroscopic analysis, ^1H NMR ($CDCl_3$, 400 MHz, δ p.p.m.): 5.16 (s, 2H, –CH_2O–), 6.51 (s, 1H, enol CH), 7.07 (d, J=8.8 Hz, 2H, Ar—H), 7.40–7.43 (m, 5H, Ar—H), 7.94 (d, J=9.2 Hz, 2H, Ar—H), 15.41 (brs, 1H, enol OH); IR (KBr, ν cm^{-1}): 1600 (C═O), 1508 (C═C), 2930 (C—H, alkyl).

Refinement

The CF_3 group was found to be disordered over two orientations. The occupancies of the disordered positions of F1/F1', F2/F2' and F3/F3' were refined to 0.641 (11) /0.359 (11). Similarity restraints with standard uncertainty of 0.01 were applied for C—F and F···F distances and the displacement parameters of the F atoms were restrained to isotopic behavior. The H atom of the hydroxyl group was located in a difference Fourier map and its position was refined freely, with U_{iso}(H) = 1.5 U_{eq}(O). Other H atoms were refined as riding, with C—H = 0.93 to 0.97 Å, and with U_{iso}(H) = 1.2 (1.5 for methyl) U_{eq}(C). In the absence of significant anomalous scattering effects Friedel pairs have been merged.

Figures

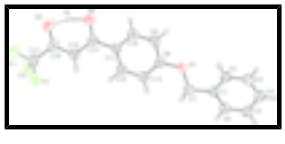


Fig. 1. The molecular structure of (I), showing the atom-labeling scheme. Displacement ellipsoids of non-H atoms are drawn at the 30% probability level. Only one component of the disordered CF_3 group is shown.

4-[4-(Benzyloxy)phenyl]-1,1,1-trifluoro-4-hydroxybut-3-en-2-one

Crystal data

$C_{17}H_{13}F_3O_3$	F_{000} = 664
M_r = 322.27	D_x = 1.429 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 923 reflections
a = 7.6226 (11) Å	θ = 2.3–19.1°
b = 11.0708 (15) Å	μ = 0.12 mm^{-1}
c = 17.748 (2) Å	T = 294 (2) K
V = 1497.7 (3) Å^3	Block, colorless
Z = 4	0.20 × 0.10 × 0.10 mm

Data collection

Bruker APEX CCD area-detector diffractometer	1881 independent reflections
Radiation source: fine-focus sealed tube	1110 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.044
T = 294(2) K	θ_{max} = 27.0°
φ and ω scans	θ_{min} = 2.2°
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)	h = −9→9
T_{min} = 0.976, T_{max} = 0.983	k = −14→12
6737 measured reflections	l = −16→22

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.057	$w = 1/[\sigma^2(F_o^2) + (0.0746P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.152	$(\Delta/\sigma)_{max} < 0.001$
S = 1.06	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
1881 reflections	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
239 parameters	Extinction correction: none
66 restraints	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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 ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.3740 (6)	0.8855 (4)	−0.1935 (2)	0.0626 (12)	
C2	0.3147 (6)	0.7911 (4)	−0.2368 (3)	0.0690 (12)	
H2	0.2789	0.7200	−0.2136	0.083*	
C3	0.3078 (7)	0.8003 (5)	−0.3137 (3)	0.0787 (14)	
H3	0.2660	0.7360	−0.3422	0.094*	
C4	0.3626 (7)	0.9049 (5)	−0.3489 (3)	0.0815 (15)	
H4	0.3585	0.9109	−0.4011	0.098*	
C5	0.4231 (7)	1.0001 (5)	−0.3067 (3)	0.0835 (15)	
H5	0.4582	1.0711	−0.3302	0.100*	
C6	0.4317 (6)	0.9904 (4)	−0.2297 (3)	0.0714 (12)	
H6	0.4762	1.0541	−0.2014	0.086*	
C7	0.3784 (7)	0.8811 (4)	−0.1086 (2)	0.0668 (12)	
H7A	0.4802	0.9245	−0.0901	0.080*	
H7B	0.2741	0.9192	−0.0882	0.080*	
C8	0.3801 (6)	0.7331 (4)	−0.0104 (2)	0.0612 (11)	
C9	0.3715 (7)	0.6124 (4)	0.0080 (3)	0.0793 (14)	
H9	0.3680	0.5548	−0.0301	0.095*	
C10	0.3680 (7)	0.5765 (4)	0.0812 (2)	0.0742 (14)	
H10	0.3623	0.4945	0.0924	0.089*	
C11	0.3728 (6)	0.6596 (3)	0.1395 (2)	0.0561 (10)	
C12	0.3821 (7)	0.7810 (4)	0.1203 (2)	0.0683 (13)	
H12	0.3847	0.8387	0.1584	0.082*	
C13	0.3876 (6)	0.8183 (4)	0.0466 (2)	0.0691 (13)	
H13	0.3962	0.9000	0.0350	0.083*	
C14	0.3696 (5)	0.6193 (4)	0.2183 (2)	0.0620 (11)	
C15	0.3426 (6)	0.6954 (4)	0.2797 (2)	0.0626 (11)	
H15	0.3231	0.7773	0.2719	0.075*	
C16	0.3446 (6)	0.6505 (4)	0.3516 (3)	0.0678 (13)	
C17	0.3182 (7)	0.7320 (5)	0.4185 (3)	0.0852 (16)	
F1	0.2753 (14)	0.6788 (5)	0.4809 (3)	0.122 (3)	0.641 (11)
F2	0.1903 (12)	0.8142 (7)	0.4089 (3)	0.122 (3)	0.641 (11)
F3	0.4573 (10)	0.7974 (9)	0.4330 (5)	0.141 (4)	0.641 (11)
F1'	0.436 (2)	0.6954 (13)	0.4677 (8)	0.151 (6)	0.359 (11)

F2'	0.1678 (14)	0.7131 (15)	0.4486 (9)	0.142 (6)	0.359 (11)
F3'	0.343 (3)	0.8437 (7)	0.4061 (7)	0.120 (5)	0.359 (11)
O1	0.3861 (5)	0.7590 (2)	−0.08524 (15)	0.0738 (10)	
O2	0.3947 (5)	0.5056 (3)	0.2297 (2)	0.0887 (11)	
O3	0.3710 (6)	0.5400 (3)	0.3683 (2)	0.0963 (12)	
H1	0.370 (9)	0.503 (5)	0.300 (4)	0.144*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.063 (3)	0.068 (3)	0.057 (3)	0.008 (3)	0.003 (2)	0.002 (2)
C2	0.072 (3)	0.067 (3)	0.068 (3)	0.000 (2)	−0.006 (2)	0.002 (2)
C3	0.088 (4)	0.076 (3)	0.072 (3)	−0.002 (3)	−0.009 (3)	−0.005 (3)
C4	0.084 (4)	0.097 (4)	0.063 (3)	0.006 (3)	−0.007 (3)	0.010 (3)
C5	0.095 (4)	0.084 (3)	0.071 (3)	0.006 (3)	0.007 (3)	0.020 (3)
C6	0.082 (3)	0.061 (3)	0.071 (3)	−0.002 (2)	0.006 (3)	0.003 (2)
C7	0.079 (3)	0.060 (3)	0.062 (3)	−0.001 (3)	−0.002 (3)	−0.003 (2)
C8	0.065 (3)	0.058 (2)	0.061 (3)	0.004 (2)	0.004 (2)	0.001 (2)
C9	0.110 (4)	0.060 (3)	0.068 (3)	−0.006 (3)	0.007 (3)	−0.010 (2)
C10	0.101 (4)	0.050 (2)	0.072 (3)	0.005 (3)	0.008 (3)	−0.003 (2)
C11	0.057 (3)	0.055 (2)	0.056 (2)	0.003 (2)	0.001 (2)	−0.0025 (19)
C12	0.094 (4)	0.055 (3)	0.056 (3)	−0.007 (3)	0.004 (3)	−0.004 (2)
C13	0.087 (3)	0.052 (2)	0.069 (3)	−0.004 (3)	−0.002 (3)	−0.001 (2)
C14	0.057 (3)	0.053 (2)	0.076 (3)	0.006 (2)	0.005 (3)	0.011 (2)
C15	0.074 (3)	0.056 (2)	0.058 (3)	−0.005 (2)	0.004 (2)	0.004 (2)
C16	0.069 (3)	0.068 (3)	0.066 (3)	−0.006 (2)	−0.003 (3)	0.015 (2)
C17	0.104 (5)	0.088 (4)	0.064 (3)	−0.011 (4)	−0.017 (3)	0.012 (3)
F1	0.186 (8)	0.106 (4)	0.075 (4)	0.007 (5)	0.043 (4)	0.026 (3)
F2	0.171 (7)	0.117 (5)	0.079 (4)	0.047 (5)	−0.025 (4)	−0.024 (3)
F3	0.130 (6)	0.153 (7)	0.141 (6)	−0.061 (5)	0.021 (5)	−0.054 (5)
F1'	0.172 (10)	0.180 (11)	0.100 (8)	−0.008 (9)	−0.064 (7)	−0.014 (7)
F2'	0.120 (8)	0.173 (11)	0.134 (10)	−0.023 (8)	0.035 (7)	−0.046 (8)
F3'	0.160 (11)	0.091 (7)	0.111 (7)	−0.017 (7)	0.001 (8)	−0.003 (5)
O1	0.107 (3)	0.0595 (18)	0.0547 (18)	0.0060 (19)	0.0085 (18)	0.0011 (14)
O2	0.122 (3)	0.060 (2)	0.084 (2)	0.011 (2)	0.020 (2)	0.0129 (16)
O3	0.126 (3)	0.086 (2)	0.077 (2)	0.016 (2)	0.012 (2)	0.0271 (19)

Geometric parameters (Å, °)

C1—C2	1.373 (6)	C10—H10	0.9300
C1—C6	1.398 (6)	C11—C12	1.389 (6)
C1—C7	1.508 (6)	C11—C14	1.467 (6)
C2—C3	1.369 (6)	C12—C13	1.373 (6)
C2—H2	0.9300	C12—H12	0.9300
C3—C4	1.380 (7)	C13—H13	0.9300
C3—H3	0.9300	C14—O2	1.289 (5)
C4—C5	1.372 (7)	C14—C15	1.394 (6)
C4—H4	0.9300	C15—C16	1.369 (6)
C5—C6	1.372 (6)	C15—H15	0.9300

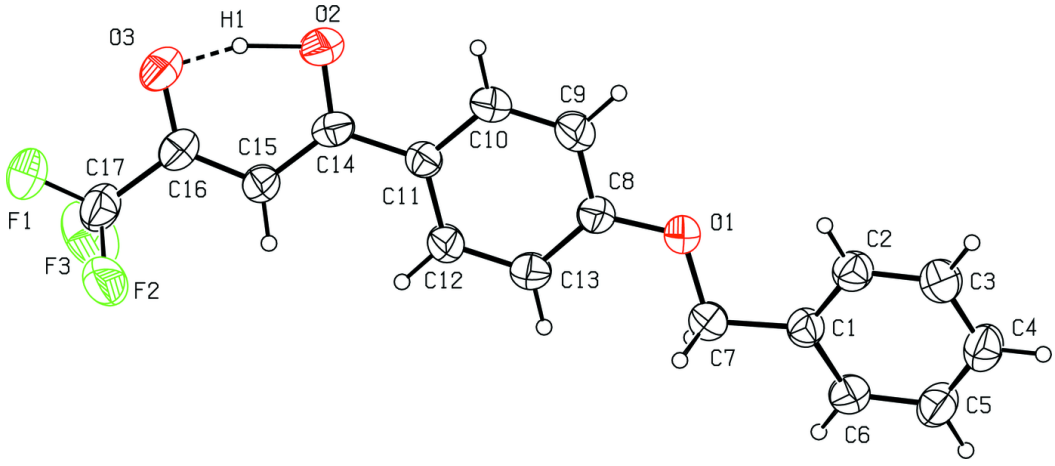
C5—H5	0.9300	C16—O3	1.274 (5)
C6—H6	0.9300	C16—C17	1.505 (7)
C7—O1	1.415 (5)	C17—F3'	1.270 (7)
C7—H7A	0.9700	C17—F2'	1.282 (7)
C7—H7B	0.9700	C17—F1	1.296 (6)
C8—O1	1.360 (5)	C17—F3	1.309 (6)
C8—C9	1.377 (6)	C17—F1'	1.318 (7)
C8—C13	1.384 (6)	C17—F2	1.344 (6)
C9—C10	1.359 (6)	O2—H1	1.26 (6)
C9—H9	0.9300	O3—H1	1.29 (6)
C10—C11	1.385 (6)		
C2—C1—C6	118.6 (4)	C10—C11—C14	120.6 (4)
C2—C1—C7	122.8 (4)	C12—C11—C14	122.0 (4)
C6—C1—C7	118.6 (4)	C13—C12—C11	121.8 (4)
C3—C2—C1	120.9 (4)	C13—C12—H12	119.1
C3—C2—H2	119.5	C11—C12—H12	119.1
C1—C2—H2	119.5	C12—C13—C8	119.4 (4)
C2—C3—C4	120.1 (5)	C12—C13—H13	120.3
C2—C3—H3	119.9	C8—C13—H13	120.3
C4—C3—H3	119.9	O2—C14—C15	119.3 (4)
C5—C4—C3	119.9 (5)	O2—C14—C11	116.4 (4)
C5—C4—H4	120.0	C15—C14—C11	124.3 (4)
C3—C4—H4	120.0	C16—C15—C14	120.5 (4)
C6—C5—C4	120.0 (5)	C16—C15—H15	119.7
C6—C5—H5	120.0	C14—C15—H15	119.7
C4—C5—H5	120.0	O3—C16—C15	124.5 (5)
C5—C6—C1	120.5 (5)	O3—C16—C17	114.4 (4)
C5—C6—H6	119.8	C15—C16—C17	121.0 (4)
C1—C6—H6	119.8	F3'—C17—F2'	111.4 (7)
O1—C7—C1	108.9 (3)	F1—C17—F3	106.7 (5)
O1—C7—H7A	109.9	F3'—C17—F1'	108.3 (7)
C1—C7—H7A	109.9	F2'—C17—F1'	106.4 (7)
O1—C7—H7B	109.9	F1—C17—F2	103.5 (5)
C1—C7—H7B	109.9	F3—C17—F2	103.8 (5)
H7A—C7—H7B	108.3	F3'—C17—C16	115.4 (7)
O1—C8—C9	116.0 (4)	F2'—C17—C16	110.5 (7)
O1—C8—C13	124.6 (4)	F1—C17—C16	115.8 (5)
C9—C8—C13	119.4 (4)	F3—C17—C16	112.2 (5)
C10—C9—C8	120.8 (4)	F1'—C17—C16	104.3 (7)
C10—C9—H9	119.6	F2—C17—C16	113.8 (4)
C8—C9—H9	119.6	C8—O1—C7	119.1 (3)
C9—C10—C11	121.3 (4)	C14—O2—H1	99 (3)
C9—C10—H10	119.4	C16—O3—H1	95 (3)
C11—C10—H10	119.4	C16—O3—H1	95 (3)
C10—C11—C12	117.4 (4)		
C6—C1—C2—C3	−1.7 (7)	C12—C11—C14—C15	12.5 (7)
C7—C1—C2—C3	177.9 (5)	O2—C14—C15—C16	0.7 (7)
C1—C2—C3—C4	0.8 (8)	C11—C14—C15—C16	−178.9 (4)

C2—C3—C4—C5	−0.4 (8)	C14—C15—C16—O3	0.3 (8)
C3—C4—C5—C6	1.1 (8)	C14—C15—C16—C17	179.1 (4)
C4—C5—C6—C1	−2.0 (8)	O3—C16—C17—F3'	160.2 (10)
C2—C1—C6—C5	2.3 (7)	C15—C16—C17—F3'	−18.7 (11)
C7—C1—C6—C5	−177.3 (5)	O3—C16—C17—F2'	−72.4 (11)
C2—C1—C7—O1	24.2 (7)	C15—C16—C17—F2'	108.7 (11)
C6—C1—C7—O1	−156.2 (4)	O3—C16—C17—F1	−19.0 (8)
O1—C8—C9—C10	178.8 (4)	C15—C16—C17—F1	162.1 (7)
C13—C8—C9—C10	0.8 (9)	O3—C16—C17—F3	103.8 (8)
C8—C9—C10—C11	0.1 (9)	C15—C16—C17—F3	−75.2 (8)
C9—C10—C11—C12	−0.3 (8)	O3—C16—C17—F1'	41.6 (11)
C9—C10—C11—C14	−179.8 (5)	C15—C16—C17—F1'	−137.3 (10)
C10—C11—C12—C13	−0.4 (8)	O3—C16—C17—F2	−138.7 (7)
C14—C11—C12—C13	179.1 (4)	C15—C16—C17—F2	42.3 (8)
C11—C12—C13—C8	1.3 (8)	C9—C8—O1—C7	174.1 (4)
O1—C8—C13—C12	−179.3 (4)	C13—C8—O1—C7	−8.0 (8)
C9—C8—C13—C12	−1.5 (8)	C1—C7—O1—C8	−175.9 (4)
C10—C11—C14—O2	12.3 (7)	C15—C16—O3—H1	−7(3)
C12—C11—C14—O2	−167.1 (5)	C17—C16—O3—H1	174 (3)
C10—C11—C14—C15	−168.0 (5)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H1···O3	1.26 (6)	1.29 (6)	2.496 (5)	158 (5)

Fig. 1


H1
O2
O3
C9
C10
C15
C14
C17
C16
C11
C8
O1
C2
C3
F1
F3
F2
C12
C13
C1
C4
C7
C5
C6