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(*S*)-Benzyl 2-(6-methyl-2-pyridylaminocarbonyl)pyrrolidine-1-carboxylate

Tai-Ran Kang,[a]* Seik Weng Ng[b] and Long He[a]

[a]College of Chemistry and Chemical Engineering, China West Normal University, Nanchong 637002, People's Republic of China, and [b]Department of Chemistry, University of Malaya, Kuala Lumpur 50603, Malaysia
Correspondence e-mail: cwnuchem@163.com

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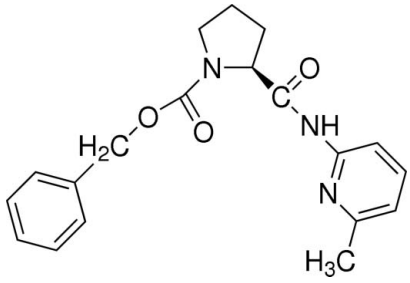
Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.092; data-to-parameter ratio = 9.7.

The molecules of the title compound, $C_{19}H_{21}N_3O_3$, are linked by a hydrogen bond between the amide NH group and the ester substituent in the 1-position of the pyrrolidine ring. This gives rise to a helical chain that propagates along the *a* axis of the orthorhombic cell by a 2_1 screw translation.

Related literature

For benzyl (*S*)-2-(5-methyl-2-pyridylaminocarbonyl)pyrrolidine-2-carboxylate, see He (2006).



Experimental

Crystal data

$C_{19}H_{21}N_3O_3$
M_r = 339.39
Orthorhombic, $P2_12_12_1$
a = 6.9065 (1) Å
b = 15.7480 (4) Å
c = 15.8380 (4) Å
V = 1722.60 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 153 (2) K
0.45 × 0.44 × 0.22 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.961, T_{max} = 0.981
16810 measured reflections
2261 independent reflections
2207 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.092
S = 1.04
2261 reflections
232 parameters
8 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2^{i}	0.88 (1)	2.18 (1)	3.036 (2)	163 (2)

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2028).

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supplementary materials

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(*S*)-Benzyl 2-(6-methyl-2-pyridylaminocarbonyl)pyrrolidine-1-carboxylate

T.-R. Kang, S. W. Ng and L. He

Comment

A recent study of benzyl (*S*)-2-(5-methyl-2-pyridylaminocarbonyl)-pyrrolidine-1-carboxylate shows a hydrogen-bonded chain [N···O 2.927 (2) Å] that runs along *b*-axis [11.4256 (3) Å] of the orthorhombic unit cell. In the isomeric title compound, the molecules are linked by the corresponding amido-carbonyl hydrogen bond [N···O 3.036 (2) Å] into a helical chain that runs along the *a*-axis of the orthorhombic cell. The mode of propagation differs between the two, as noted from the pitch of the helix.

Experimental

The procedure for synthesizing benzyl (*S*)-2-(5-methyl-2-pyridylaminocarbonyl)-pyrrolidine-1-carboxylate (He, 2006) was used, with 6-methylpyridyl-2-amine in place of 5-methylpyridyl-2-amine. Crystals were grown from an ethanolic solution of the compound.

Refinement

The carbon-bound hydrogen atoms were placed at calculated positions (C–H 0.95 to 0.99 Å) and were inlcuded in the refinement in the riding model approximation, with *U*(H) set to 1.2 to 1.5 U_{eq}(C). The amido hydrogen atom was located in a difference Fourier map and was refined with a distance restraint of N–H 0.88 ± 0.01 Å; its temperature factor was freely refined.

Figures

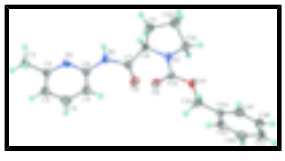


Fig. 1. Thermal ellipsoid plot of $C_{19}H_{21}N_3O_3$. Displacement ellipsoids are drawn at the 70% probability level, H atoms are drawn as spheres of arbitrary radiui.

(*S*)-Benzyl 2-(6-methyl-2-pyridylaminocarbonyl)pyrrolidine-1-carboxylate

Crystal data

$C_{19}H_{21}N_3O_3$	F_{000} = 720
M_r = 339.39	D_x = 1.309 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 16166 reflections
a = 6.9065 (1) Å	θ = 3.2–27.5º

b = 15.7480 (4) Å	μ = 0.09 mm^{-1}
c = 15.8380 (4) Å	T = 153 (2) K
V = 1722.60 (7) Å^3	Block, colorless
Z = 4	0.45 × 0.44 × 0.22 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer	2261 independent reflections
Radiation source: rotating anode tube	2207 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.019
T = 153(2) K	θ_{max} = 27.5º
ω scans	θ_{min} = 3.2º
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	h = −8→8
T_{min} = 0.961, T_{max} = 0.981	k = −20→20
16810 measured reflections	l = −20→20

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0611P)^2 + 0.4025P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.032	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.092	$\Delta\rho_{max}$ = 0.53 e Å^{-3}
S = 1.04	$\Delta\rho_{min}$ = −0.20 e Å^{-3}
2261 reflections	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
232 parameters	Extinction coefficient: 0.033 (3)
8 restraints	Absolute structure: Friedel pairs were merged
Primary atom site location: structure-invariant direct methods	Flack parameter: 0
Secondary atom site location: difference Fourier map	Rogers parameter: ?
Hydrogen site location: inferred from neighbouring sites	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.9391 (2)	0.09533 (8)	0.58395 (7)	0.0265 (3)
O2	0.91177 (19)	0.29212 (8)	0.61477 (7)	0.0234 (3)
O3	1.00096 (19)	0.27940 (8)	0.75267 (7)	0.0226 (3)
N1	0.9295 (2)	0.09490 (8)	0.31762 (8)	0.0196 (3)
N2	1.0429 (2)	0.12574 (9)	0.44994 (8)	0.0198 (3)
H2	1.134 (3)	0.1534 (12)	0.4225 (13)	0.032 (6)*
N3	1.1779 (2)	0.21679 (9)	0.65344 (8)	0.0196 (3)
C1	0.8381 (3)	0.07319 (13)	0.17138 (10)	0.0289 (4)

H1A	0.9745	0.0672	0.1554	0.043*
H1B	0.7958	0.1317	0.1611	0.043*
H1C	0.7592	0.0342	0.1376	0.043*
C2	0.8148 (2)	0.05246 (10)	0.26361 (10)	0.0215 (3)
C3	0.6786 (3)	−0.00678 (11)	0.29092 (11)	0.0254 (4)
H3	0.6003	−0.0365	0.2515	0.030*
C4	0.6600 (3)	−0.02138 (11)	0.37685 (12)	0.0277 (4)
H4	0.5674	−0.0612	0.3969	0.033*
C5	0.7765 (3)	0.02218 (11)	0.43353 (10)	0.0245 (4)
H5	0.7653	0.0134	0.4927	0.029*
C6	0.9107 (2)	0.07922 (9)	0.40043 (10)	0.0188 (3)
C7	1.0491 (2)	0.13158 (9)	0.53608 (9)	0.0183 (3)
C8	1.2178 (2)	0.18583 (10)	0.56818 (9)	0.0183 (3)
H8	1.2434	0.2343	0.5289	0.022*
C9	1.4007 (3)	0.13120 (11)	0.57953 (11)	0.0249 (4)
H9A	1.3667	0.0703	0.5837	0.030*
H9B	1.4907	0.1392	0.5315	0.030*
C10	1.4912 (3)	0.16248 (13)	0.66126 (11)	0.0301 (4)
H10A	1.5743	0.2127	0.6511	0.036*
H10B	1.5698	0.1174	0.6881	0.036*
C11	1.3167 (3)	0.18557 (11)	0.71607 (10)	0.0239 (4)
H11A	1.2658	0.1353	0.7463	0.029*
H11B	1.3499	0.2302	0.7576	0.029*
C12	1.0218 (2)	0.26492 (10)	0.66888 (9)	0.0181 (3)
C13	0.8416 (3)	0.33562 (11)	0.77456 (10)	0.0251 (4)
H13A	0.7232	0.3188	0.7436	0.030*
H13B	0.8739	0.3949	0.7591	0.030*
C14	0.8084 (2)	0.32899 (10)	0.86799 (10)	0.0193 (3)
C15	0.7404 (3)	0.25312 (11)	0.90256 (11)	0.0242 (3)
H15	0.7205	0.2052	0.8671	0.029*
C16	0.7017 (3)	0.24711 (14)	0.98825 (12)	0.0309 (4)
H16	0.6578	0.1950	1.0116	0.037*
C17	0.7273 (3)	0.31743 (15)	1.03973 (11)	0.0344 (5)
H17	0.6986	0.3136	1.0983	0.041*
C18	0.7942 (3)	0.39275 (13)	1.00651 (12)	0.0322 (4)
H18	0.8111	0.4408	1.0420	0.039*
C19	0.8372 (3)	0.39844 (11)	0.92059 (11)	0.0250 (3)
H19	0.8863	0.4500	0.8980	0.030*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0286 (6)	0.0308 (6)	0.0200 (5)	−0.0078 (6)	0.0056 (5)	0.0008 (5)
O2	0.0228 (6)	0.0309 (6)	0.0167 (5)	0.0053 (5)	−0.0015 (5)	0.0009 (4)
O3	0.0220 (5)	0.0312 (6)	0.0145 (5)	0.0091 (5)	−0.0007 (4)	−0.0024 (4)
N1	0.0188 (6)	0.0221 (6)	0.0179 (6)	−0.0021 (6)	−0.0003 (5)	−0.0033 (5)
N2	0.0201 (7)	0.0229 (6)	0.0163 (6)	−0.0061 (5)	0.0015 (5)	0.0002 (5)
N3	0.0199 (7)	0.0260 (6)	0.0129 (5)	0.0038 (6)	−0.0018 (5)	−0.0020 (5)

C1	0.0268 (9)	0.0396 (9)	0.0204 (7)	−0.0062 (8)	−0.0010 (7)	−0.0081 (7)
C2	0.0170 (7)	0.0251 (7)	0.0224 (7)	0.0008 (7)	−0.0003 (6)	−0.0068 (6)
C3	0.0204 (8)	0.0257 (7)	0.0299 (8)	−0.0041 (7)	−0.0022 (7)	−0.0079 (7)
C4	0.0224 (9)	0.0256 (8)	0.0352 (9)	−0.0076 (7)	0.0029 (7)	−0.0013 (7)
C5	0.0246 (8)	0.0258 (8)	0.0230 (7)	−0.0052 (7)	0.0016 (7)	0.0011 (7)
C6	0.0176 (7)	0.0181 (6)	0.0207 (7)	0.0001 (6)	0.0006 (6)	−0.0024 (5)
C7	0.0198 (8)	0.0178 (6)	0.0174 (7)	0.0007 (6)	0.0002 (6)	0.0009 (5)
C8	0.0197 (7)	0.0221 (7)	0.0131 (6)	−0.0003 (6)	0.0007 (6)	−0.0016 (5)
C9	0.0206 (8)	0.0313 (8)	0.0227 (7)	0.0052 (7)	0.0012 (7)	−0.0030 (7)
C10	0.0258 (9)	0.0345 (9)	0.0301 (9)	0.0048 (8)	−0.0021 (8)	−0.0008 (7)
C11	0.0250 (8)	0.0313 (8)	0.0155 (7)	0.0069 (7)	−0.0044 (7)	0.0004 (6)
C12	0.0185 (7)	0.0209 (6)	0.0150 (6)	−0.0008 (6)	−0.0001 (6)	−0.0004 (5)
C13	0.0247 (8)	0.0308 (8)	0.0198 (7)	0.0110 (7)	0.0020 (7)	0.0008 (6)
C14	0.0141 (7)	0.0247 (7)	0.0189 (7)	0.0043 (6)	−0.0004 (6)	−0.0013 (6)
C15	0.0215 (8)	0.0259 (8)	0.0252 (8)	−0.0020 (7)	−0.0026 (7)	−0.0033 (6)
C16	0.0215 (8)	0.0409 (9)	0.0302 (9)	−0.0044 (8)	0.0016 (7)	0.0095 (8)
C17	0.0240 (9)	0.0612 (13)	0.0180 (8)	0.0038 (9)	0.0023 (7)	−0.0025 (8)
C18	0.0266 (9)	0.0422 (10)	0.0277 (8)	0.0050 (8)	−0.0014 (8)	−0.0162 (8)
C19	0.0212 (8)	0.0235 (7)	0.0302 (8)	0.0010 (7)	0.0009 (7)	−0.0044 (7)

Geometric parameters (Å, °)

O1—C7	1.216 (2)	C8—C9	1.539 (2)
O2—C12	1.223 (2)	C8—H8	1.0000
O3—C12	1.3541 (18)	C9—C10	1.520 (2)
O3—C13	1.455 (2)	C9—H9A	0.9900
N1—C6	1.341 (2)	C9—H9B	0.9900
N1—C2	1.344 (2)	C10—C11	1.530 (2)
N2—C7	1.3682 (19)	C10—H10A	0.9900
N2—C6	1.409 (2)	C10—H10B	0.9900
N2—H2	0.880 (10)	C11—H11A	0.9900
N3—C12	1.341 (2)	C11—H11B	0.9900
N3—C8	1.4618 (18)	C13—C14	1.501 (2)
N3—C11	1.4642 (19)	C13—H13A	0.9900
C1—C2	1.505 (2)	C13—H13B	0.9900
C1—H1A	0.9800	C14—C19	1.389 (2)
C1—H1B	0.9800	C14—C15	1.396 (2)
C1—H1C	0.9800	C15—C16	1.386 (2)
C2—C3	1.393 (2)	C15—H15	0.9500
C3—C4	1.386 (2)	C16—C17	1.387 (3)
C3—H3	0.9500	C16—H16	0.9500
C4—C5	1.387 (2)	C17—C18	1.377 (3)
C4—H4	0.9500	C17—H17	0.9500
C5—C6	1.393 (2)	C18—C19	1.396 (2)
C5—H5	0.9500	C18—H18	0.9500
C7—C8	1.531 (2)	C19—H19	0.9500
C12—O3—C13	114.62 (13)	C8—C9—H9B	110.8
C6—N1—C2	118.28 (14)	H9A—C9—H9B	108.9
C7—N2—C6	127.62 (14)	C9—C10—C11	103.66 (14)

C7—N2—H2	115.9 (15)	C9—C10—H10A	111.0
C6—N2—H2	116.4 (15)	C11—C10—H10A	111.0
C12—N3—C8	120.57 (13)	C9—C10—H10B	111.0
C12—N3—C11	126.34 (13)	C11—C10—H10B	111.0
C8—N3—C11	112.99 (13)	H10A—C10—H10B	109.0
C2—C1—H1A	109.5	N3—C11—C10	102.19 (13)
C2—C1—H1B	109.5	N3—C11—H11A	111.3
H1A—C1—H1B	109.5	C10—C11—H11A	111.3
C2—C1—H1C	109.5	N3—C11—H11B	111.3
H1A—C1—H1C	109.5	C10—C11—H11B	111.3
H1B—C1—H1C	109.5	H11A—C11—H11B	109.2
N1—C2—C3	122.23 (15)	O2—C12—N3	124.75 (14)
N1—C2—C1	116.52 (15)	O2—C12—O3	124.17 (15)
C3—C2—C1	121.24 (15)	N3—C12—O3	111.08 (13)
C4—C3—C2	118.59 (16)	O3—C13—C14	107.95 (13)
C4—C3—H3	120.7	O3—C13—H13A	110.1
C2—C3—H3	120.7	C14—C13—H13A	110.1
C3—C4—C5	119.96 (17)	O3—C13—H13B	110.1
C3—C4—H4	120.0	C14—C13—H13B	110.1
C5—C4—H4	120.0	H13A—C13—H13B	108.4
C4—C5—C6	117.47 (16)	C19—C14—C15	119.13 (15)
C4—C5—H5	121.3	C19—C14—C13	120.97 (16)
C6—C5—H5	121.3	C15—C14—C13	119.85 (15)
N1—C6—C5	123.45 (15)	C16—C15—C14	120.49 (17)
N1—C6—N2	112.70 (14)	C16—C15—H15	119.8
C5—C6—N2	123.84 (14)	C14—C15—H15	119.8
O1—C7—N2	124.79 (15)	C15—C16—C17	119.77 (19)
O1—C7—C8	122.04 (14)	C15—C16—H16	120.1
N2—C7—C8	113.10 (14)	C17—C16—H16	120.1
N3—C8—C7	110.45 (13)	C18—C17—C16	120.39 (16)
N3—C8—C9	103.48 (12)	C18—C17—H17	119.8
C7—C8—C9	110.57 (13)	C16—C17—H17	119.8
N3—C8—H8	110.7	C17—C18—C19	119.94 (17)
C7—C8—H8	110.7	C17—C18—H18	120.0
C9—C8—H8	110.7	C19—C18—H18	120.0
C10—C9—C8	104.84 (13)	C14—C19—C18	120.25 (16)
C10—C9—H9A	110.8	C14—C19—H19	119.9
C8—C9—H9A	110.8	C18—C19—H19	119.9
C10—C9—H9B	110.8		
C6—N1—C2—C3	−0.1 (2)	C7—C8—C9—C10	−139.34 (14)
C6—N1—C2—C1	178.90 (15)	C8—C9—C10—C11	34.49 (18)
N1—C2—C3—C4	0.8 (3)	C12—N3—C11—C10	−161.55 (16)
C1—C2—C3—C4	−178.13 (17)	C8—N3—C11—C10	21.96 (18)
C2—C3—C4—C5	−0.5 (3)	C9—C10—C11—N3	−33.99 (18)
C3—C4—C5—C6	−0.5 (3)	C8—N3—C12—O2	−6.1 (2)
C2—N1—C6—C5	−1.0 (2)	C11—N3—C12—O2	177.70 (16)
C2—N1—C6—N2	178.20 (14)	C8—N3—C12—O3	173.69 (14)
C4—C5—C6—N1	1.3 (3)	C11—N3—C12—O3	−2.6 (2)
C4—C5—C6—N2	−177.80 (16)	C13—O3—C12—O2	−3.9 (2)

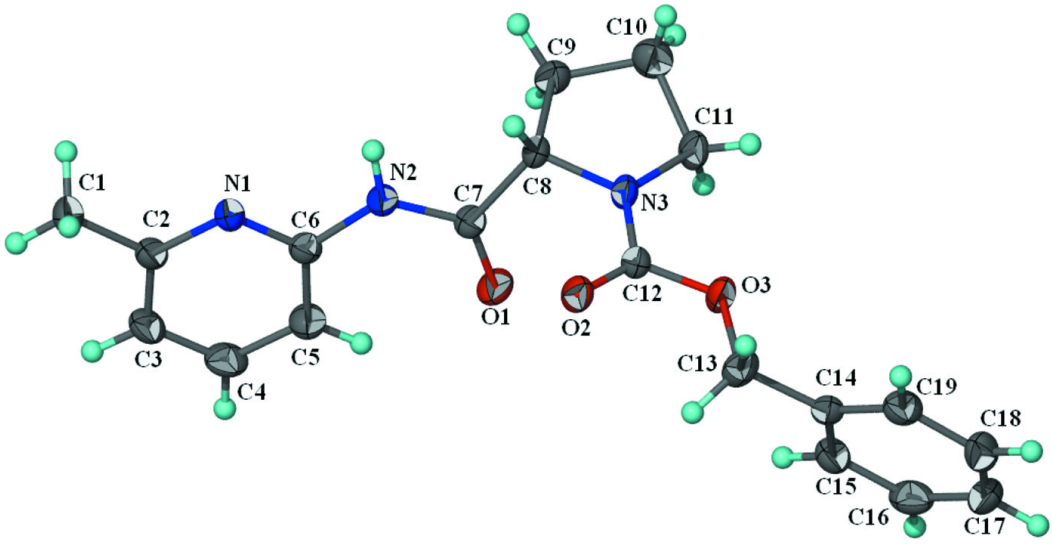
C7—N2—C6—N1	172.46 (15)	C13—O3—C12—N3	176.36 (14)
C7—N2—C6—C5	−8.4 (3)	C12—O3—C13—C14	166.87 (14)
C6—N2—C7—O1	1.0 (3)	O3—C13—C14—C19	115.82 (17)
C6—N2—C7—C8	178.08 (14)	O3—C13—C14—C15	−66.7 (2)
C12—N3—C8—C7	−59.16 (18)	C19—C14—C15—C16	0.1 (3)
C11—N3—C8—C7	117.55 (15)	C13—C14—C15—C16	−177.40 (17)
C12—N3—C8—C9	−177.53 (14)	C14—C15—C16—C17	1.2 (3)
C11—N3—C8—C9	−0.81 (18)	C15—C16—C17—C18	−1.1 (3)
O1—C7—C8—N3	−24.6 (2)	C16—C17—C18—C19	−0.2 (3)
N2—C7—C8—N3	158.28 (13)	C15—C14—C19—C18	−1.5 (3)
O1—C7—C8—C9	89.38 (19)	C13—C14—C19—C18	176.02 (17)
N2—C7—C8—C9	−87.78 (16)	C17—C18—C19—C14	1.5 (3)
N3—C8—C9—C10	−21.06 (17)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2^{i}	0.88 (1)	2.18 (1)	3.036 (2)	163 (2)

Symmetry codes: (i) *x*+1/2, −*y*+1/2, −*z*+1.

Fig. 1


C10
C9
C11
C1
N1
N2
C7
C8
N3
C2
C6
O1
O2
C12
O3
C3
C5
C13
C14
C19
C4
C18
C15
C16
C17