Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Tetraethylammonium tricarbonyl-chlorido(pyrazine-2-carboxylato-N^1,O)-rhenate(I)

Janine Suthiram,[a] Kanyisa Mhlaba,[a] Jan Rijn Zeevaart,[a] Hendrik G. Visser[b]* and Andreas Roodt[b]

[a]Radiochemistry, South African Nuclear Energy Corporation Ltd. (Necsa), PO Box 582, Pretoria, 0001, South Africa, and [b]Department of Chemistry, University of the Free State, PO Box 339, Bloemfontein, 9300, South Africa
Correspondence e-mail: visserhg.sci@mail.uovs.ac.za

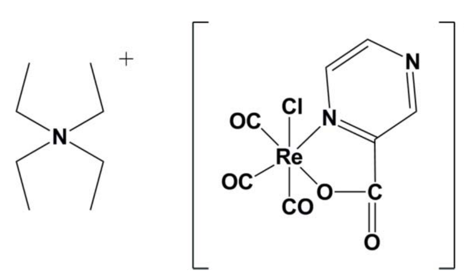
Received 12 October 2009; accepted 14 October 2009

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.006 Å; R factor = 0.021; wR factor = 0.098; data-to-parameter ratio = 20.3.

In the title complex, $(C_8H_{20}N)[Re(C_5H_3N_2O_2)Cl(CO)_3]$, the Re^I atom is coordinated facially by three carbonyl groups; the bidentate pyrazinecarboxylato ligand and a chlorine atom complete the distorted octahedral coordination.

Related literature

For synthetic background, see: Alberto *et al.* (1996). For related structures, see: Schutte *et al.* (2008); Kemp (2006); Wang *et al.* (2003); Alvarez *et al.* (2007); Brasey *et al.* (2004); Mundwiler *et al.* (2004). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$(C_8H_{20}N)[Re(C_5H_3N_2O_2)Cl(CO)_3]$
M_r = 559.02
Monoclinic, $P2_1/c$
a = 7.927 (5) Å
b = 22.278 (5) Å
c = 10.903 (5) Å
β = 90.506 (5)°
V = 1925.4 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 6.48 mm^{-1}
T = 100 K
0.27 × 0.20 × 0.11 mm

Data collection

Bruker X8 APEXII 4K Kappa CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.273, T_{max} = 0.539
32482 measured reflections
4781 independent reflections
4121 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.098
S = 1.18
4781 reflections
235 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.05 e Å^{-3}
$\Delta\rho_{min}$ = −1.38 e Å^{-3}

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus* and *XPREP* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors thank Necsa and the UFS for funding and permission to publish this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2218).

References

Alberto, R., Schibli, R. & Schubiger, P. A. (1996). *Polyhedron*, **15**, 1079–1083.
Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Alvarez, C. M., Garcia-Rodriguez, R. & Miguel, D. (2007). *Dalton Trans.* pp. 3546–3554.
Brandenburg, K. & Putz, H. (2005). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Brasey, T., Buryak, A., Scopelliti, R. & Severin, K. (2004). *Eur. J. Inorg. Chem.* pp. 964–967.
Bruker (2004). *SAINT-Plus, XPREP* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2005). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Kemp, G. (2006). PhD thesis, University of Johannesburg, South Africa.
Mundwiler, S., Kundig, M., Ortner, K. & Alberto, R. (2004). *Dalton Trans.* pp. 1320–1328.
Schutte, M., Visser, H. G. & Roodt, A. (2008). *Acta Cryst.* E**64**, m1610–m1611.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Wang, W., Spingler, B. & Alberto, R. (2003). *Inorg. Chim. Acta*. **355**, 386–391.

supplementary materials

Acta Cryst. (2009). E**65**, m1395   [**doi:10.1107/S1600536809042160**]

Tetraethylammonium tricarbonylchlorido(pyrazine-2-carboxylato-N^1,O)rhenate(I)

J. Suthiram, K. Mhlaba, J. R. Zeevaart, H. G. Visser and A. Roodt

Comment

The title complex, (I), forms a part of an ongoing investigation of the structural and kinetic behaviour of *fac*-$Re(CO)_3$ compounds (Schutte *et al.*, 2008). It crystallized as an anionic Re^I compound and one tetraethylammonium counter ion in the asymmetric unit (Fig. 1). The Re—CO bond distances are well within the normal range (Allen *et al.*, 1987). The small bite angle O4—Re1—N1 might be a reason for the slightly distorted octahedral geometry around the metal centre. There are no classical hydrogen bonds in the structure.

Experimental

$ReCl_3(CO)_3$ (64.2 mg, 0.01 mmol) was suspended in 10 ml methanol. The solution was heated to reflux and 2-pyrazine-carboxylic acid (13.1 mg, 0.01 mmol) dissolved in *ca* 5 ml methanol was added whilst stirring. A bright yellow colour resulted on addition of the ligand to the metal. K_2CO_3 7.1 mg (0.005 mmol) was added to the solution. The reaction solution was refluxed for 6 h after which the solvent volume was decreased on a rotovapor. The MeOH solution was layered with a minimal amount of diethyl ether and left to stand in a refrigerator. After a few days yellow crystals were formed.

Refinement

The methyl, methylene and aromatic H atoms were placed in geometrically idealized positions with C—H distances = 0.96, 0.97 and 0.96 Å, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.5U_{eq}(methyl-C) and 1.2U_{eq}(methylene and aromatic-C). The highest residual electron density was located 0.93 Å from H17C and was essentially meaningless.

Figures

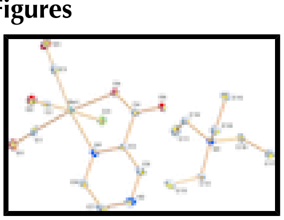


Fig. 1. A view of the title complex plotted with 50% probability displacement ellipsoids; hydrogen atoms have been omitted for clarity.

Tetraethylammonium tricarbonylchlorido(pyrazine-2-carboxylato-N^1,O)rhenate(I)

Crystal data

$(C_8H_{20}N)[Re(C_5H_3N_2O_2)Cl(CO)_3]$	F_{000} = 1088
M_r = 559.02	D_x = 1.929 Mg m^{-3}

Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 9901 reflections
a = 7.927 (5) Å	θ = 2.7–28.3°
b = 22.278 (5) Å	μ = 6.48 mm^{-1}
c = 10.903 (5) Å	T = 100 K
β = 90.506 (5)°	Cuboid, yellow
V = 1925.4 (16) Å^3	0.27 × 0.20 × 0.11 mm
Z = 4	

Data collection

Bruker X8 APEXII 4K Kappa CCD diffractometer	4781 independent reflections
Radiation source: sealed tube	4121 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.046
T = 100 K	θ_{max} = 28.3°
phi and ω scans	θ_{min} = 1.8°
Absorption correction: multi-scan (SADABS; Bruker, 2004)	$h = -10 \rightarrow 9$
T_{min} = 0.273, T_{max} = 0.539	$k = -29 \rightarrow 29$
32482 measured reflections	$l = -14 \rightarrow 13$

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0591P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.021	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.098	$\Delta\rho_{max}$ = 1.05 e Å^{-3}
S = 1.18	$\Delta\rho_{min}$ = −1.38 e Å^{-3}
4781 reflections	Extinction correction: none
235 parameters	

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.

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

	x	y	z	U_{iso}*/U_{eq}
Re1	0.30393 (2)	0.198197 (7)	0.407485 (15)	0.01064 (8)
Cl1	0.29768 (13)	0.09512 (5)	0.49378 (10)	0.0154 (2)
C5	0.3651 (5)	0.13259 (19)	0.1726 (4)	0.0117 (8)
O5	0.0904 (4)	0.10473 (15)	0.1121 (3)	0.0186 (7)

N3	0.1752 (4)	−0.07955 (16)	0.2734 (3)	0.0111 (7)
O4	0.1219 (4)	0.16145 (14)	0.2811 (3)	0.0143 (7)
N2	0.6136 (5)	0.10700 (19)	0.0608 (4)	0.0198 (9)
N1	0.4559 (5)	0.15900 (16)	0.2633 (3)	0.0132 (8)
C14	0.1057 (5)	−0.0443 (2)	0.1656 (4)	0.0146 (9)
H14B	0.1568	−0.0047	0.1666	0.018*
H14A	0.1403	−0.064	0.0906	0.018*
C4	0.1762 (5)	0.1323 (2)	0.1880 (4)	0.0131 (9)
C10	0.1294 (6)	−0.0471 (2)	0.3912 (4)	0.0169 (9)
H10A	0.1667	−0.0058	0.3849	0.02*
H10B	0.0075	−0.0467	0.3983	0.02*
C15	−0.0836 (6)	−0.0368 (2)	0.1619 (4)	0.0182 (10)
H15B	−0.1153	−0.0138	0.0909	0.027*
H15C	−0.1197	−0.0164	0.2345	0.027*
H15A	−0.1361	−0.0756	0.1578	0.027*
C12	0.3647 (5)	−0.0855 (2)	0.2602 (4)	0.0147 (9)
H12B	0.4069	−0.1106	0.3264	0.018*
H12A	0.3875	−0.1062	0.1838	0.018*
C17	0.1141 (6)	−0.1786 (2)	0.1620 (5)	0.0215 (10)
H17B	0.0637	−0.2173	0.1739	0.032*
H17A	0.231	−0.1835	0.1418	0.032*
H17C	0.0569	−0.1581	0.0962	0.032*
C1	0.4817 (6)	0.2209 (2)	0.5162 (4)	0.0160 (9)
C7	0.7019 (6)	0.1324 (2)	0.1527 (4)	0.0187 (10)
H7	0.819	0.1323	0.149	0.022*
C16	0.0997 (6)	−0.14213 (19)	0.2785 (4)	0.0139 (9)
H16A	0.1545	−0.1641	0.3445	0.017*
H16B	−0.0188	−0.1386	0.2988	0.017*
C13	0.4627 (6)	−0.0272 (2)	0.2612 (5)	0.0228 (11)
H13C	0.5808	−0.0355	0.2525	0.034*
H13B	0.4444	−0.0067	0.3374	0.034*
H13A	0.425	−0.0023	0.1945	0.034*
C8	0.6250 (6)	0.1586 (2)	0.2526 (4)	0.0167 (9)
H8	0.6913	0.1764	0.3134	0.02*
C6	0.4450 (6)	0.1077 (2)	0.0729 (4)	0.0169 (9)
H6	0.3791	0.0906	0.0111	0.02*
C11	0.2032 (6)	−0.0737 (2)	0.5073 (4)	0.0227 (11)
H11B	0.1677	−0.0504	0.5765	0.034*
H11A	0.3241	−0.0733	0.5028	0.034*
H11C	0.1646	−0.1143	0.5162	0.034*
C3	0.1409 (6)	0.2291 (2)	0.5218 (4)	0.0148 (9)
O2	0.3224 (4)	0.32482 (15)	0.2989 (3)	0.0206 (7)
O3	0.0411 (4)	0.24882 (16)	0.5855 (3)	0.0215 (7)
O1	0.5931 (4)	0.23294 (16)	0.5808 (3)	0.0241 (8)
C2	0.3140 (5)	0.2765 (2)	0.3371 (4)	0.0139 (9)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Re1	0.01130 (11)	0.00960 (12)	0.01101 (12)	−0.00026 (6)	−0.00005 (7)	−0.00013 (6)
Cl1	0.0170 (5)	0.0123 (5)	0.0169 (5)	−0.0006 (4)	−0.0002 (4)	0.0005 (4)
C5	0.013 (2)	0.010 (2)	0.012 (2)	0.0021 (16)	−0.0005 (16)	0.0015 (16)
O5	0.0130 (16)	0.0202 (19)	0.0227 (17)	−0.0029 (13)	−0.0034 (13)	−0.0071 (14)
N3	0.0113 (17)	0.0110 (18)	0.0110 (17)	−0.0010 (14)	0.0013 (13)	0.0012 (14)
O4	0.0127 (15)	0.0161 (17)	0.0141 (15)	−0.0004 (12)	−0.0006 (12)	−0.0029 (13)
N2	0.017 (2)	0.026 (2)	0.0168 (19)	0.0039 (17)	0.0031 (16)	0.0009 (17)
N1	0.0121 (17)	0.0103 (19)	0.0172 (19)	−0.0001 (14)	0.0000 (14)	0.0036 (15)
C14	0.015 (2)	0.013 (2)	0.015 (2)	0.0012 (17)	0.0014 (17)	0.0052 (17)
C4	0.011 (2)	0.013 (2)	0.015 (2)	0.0013 (16)	0.0001 (16)	0.0019 (17)
C10	0.014 (2)	0.017 (2)	0.019 (2)	0.0022 (18)	0.0026 (18)	−0.0043 (19)
C15	0.015 (2)	0.024 (3)	0.016 (2)	0.0029 (19)	−0.0025 (17)	0.0024 (19)
C12	0.010 (2)	0.017 (2)	0.018 (2)	0.0019 (17)	0.0027 (16)	−0.0006 (18)
C17	0.023 (3)	0.020 (3)	0.022 (3)	0.000 (2)	0.002 (2)	−0.005 (2)
C1	0.023 (2)	0.009 (2)	0.016 (2)	0.0009 (18)	0.0019 (18)	−0.0015 (18)
C7	0.010 (2)	0.026 (3)	0.021 (2)	0.0035 (18)	0.0040 (17)	0.006 (2)
C16	0.014 (2)	0.010 (2)	0.017 (2)	−0.0034 (17)	0.0005 (17)	0.0017 (18)
C13	0.012 (2)	0.022 (3)	0.034 (3)	−0.0006 (19)	0.000 (2)	0.004 (2)
C8	0.015 (2)	0.018 (2)	0.017 (2)	0.0012 (18)	−0.0034 (17)	0.0026 (18)
C6	0.015 (2)	0.019 (3)	0.017 (2)	−0.0002 (18)	−0.0019 (17)	0.0016 (18)
C11	0.024 (3)	0.029 (3)	0.015 (2)	0.002 (2)	−0.0004 (19)	−0.005 (2)
C3	0.018 (2)	0.012 (2)	0.015 (2)	−0.0024 (17)	−0.0025 (18)	0.0039 (18)
O2	0.0225 (18)	0.0123 (17)	0.0272 (19)	−0.0016 (14)	0.0033 (15)	0.0027 (15)
O3	0.0279 (18)	0.0198 (19)	0.0170 (17)	0.0012 (15)	0.0096 (14)	−0.0011 (14)
O1	0.0222 (18)	0.021 (2)	0.0284 (19)	0.0010 (15)	−0.0097 (15)	−0.0060 (16)
C2	0.012 (2)	0.018 (2)	0.012 (2)	0.0026 (17)	0.0006 (16)	−0.0006 (18)

Geometric parameters (Å, °)

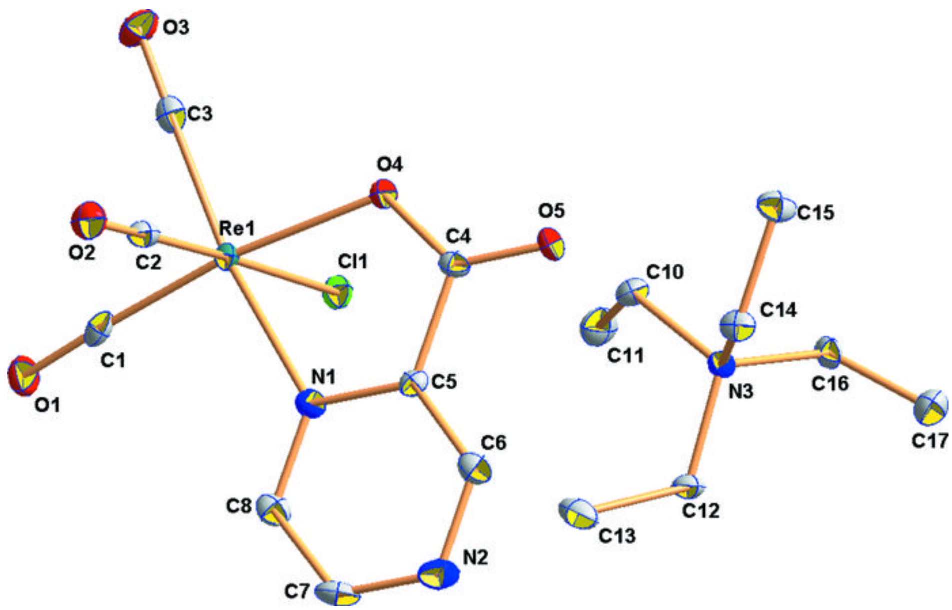
Re1—C1	1.902 (5)	C15—H15C	0.96
Re1—C2	1.908 (5)	C15—H15A	0.96
Re1—C3	1.930 (5)	C12—C13	1.514 (6)
Re1—O4	2.149 (3)	C12—H12B	0.97
Re1—N1	2.172 (4)	C12—H12A	0.97
Re1—Cl1	2.4822 (12)	C17—C16	1.513 (6)
C5—N1	1.353 (6)	C17—H17B	0.96
C5—C6	1.379 (6)	C17—H17A	0.96
C5—C4	1.508 (6)	C17—H17C	0.96
O5—C4	1.231 (5)	C1—O1	1.156 (5)
N3—C14	1.514 (5)	C7—C8	1.383 (7)
N3—C12	1.517 (5)	C7—H7	0.93
N3—C16	1.518 (5)	C16—H16A	0.97
N3—C10	1.521 (5)	C16—H16B	0.97
O4—C4	1.282 (5)	C13—H13C	0.96
N2—C7	1.342 (6)	C13—H13B	0.96

N2—C6	1.344 (6)	C13—H13A	0.96
N1—C8	1.347 (6)	C8—H8	0.93
C14—C15	1.510 (6)	C6—H6	0.93
C14—H14B	0.97	C11—H11B	0.96
C14—H14A	0.97	C11—H11A	0.96
C10—C11	1.510 (6)	C11—H11C	0.96
C10—H10A	0.97	C3—O3	1.145 (5)
C10—H10B	0.97	O2—C2	1.156 (6)
C15—H15B	0.96		

C1—Re1—C2	88.49 (19)	H15B—C15—H15C	109.5
C1—Re1—C3	89.99 (19)	C14—C15—H15A	109.5
C2—Re1—C3	87.95 (19)	H15B—C15—H15A	109.5
C1—Re1—O4	172.22 (16)	H15C—C15—H15A	109.5
C2—Re1—O4	96.95 (16)	C13—C12—N3	115.6 (4)
C3—Re1—O4	95.74 (16)	C13—C12—H12B	108.4
C1—Re1—N1	98.34 (17)	N3—C12—H12B	108.4
C2—Re1—N1	92.94 (16)	C13—C12—H12A	108.4
C3—Re1—N1	171.64 (16)	N3—C12—H12A	108.4
O4—Re1—N1	75.91 (13)	H12B—C12—H12A	107.4
C1—Re1—Cl1	91.55 (14)	C16—C17—H17B	109.5
C2—Re1—Cl1	178.07 (14)	C16—C17—H17A	109.5
C3—Re1—Cl1	93.98 (13)	H17B—C17—H17A	109.5
O4—Re1—Cl1	82.82 (9)	C16—C17—H17C	109.5
N1—Re1—Cl1	85.15 (10)	H17B—C17—H17C	109.5
N1—C5—C6	120.4 (4)	H17A—C17—H17C	109.5
N1—C5—C4	116.3 (4)	O1—C1—Re1	177.5 (4)
C6—C5—C4	123.3 (4)	N2—C7—C8	122.4 (4)
C14—N3—C12	109.0 (3)	N2—C7—H7	118.8
C14—N3—C16	111.4 (3)	C8—C7—H7	118.8
C12—N3—C16	108.3 (3)	C17—C16—N3	115.4 (4)
C14—N3—C10	108.7 (3)	C17—C16—H16A	108.4
C12—N3—C10	111.5 (3)	N3—C16—H16A	108.4
C16—N3—C10	108.0 (3)	C17—C16—H16B	108.4
C4—O4—Re1	118.2 (3)	N3—C16—H16B	108.4
C7—N2—C6	115.8 (4)	H16A—C16—H16B	107.5
C8—N1—C5	117.2 (4)	C12—C13—H13C	109.5
C8—N1—Re1	128.6 (3)	C12—C13—H13B	109.5
C5—N1—Re1	114.1 (3)	H13C—C13—H13B	109.5
C15—C14—N3	115.6 (4)	C12—C13—H13A	109.5
C15—C14—H14B	108.4	H13C—C13—H13A	109.5
N3—C14—H14B	108.4	H13B—C13—H13A	109.5
C15—C14—H14A	108.4	N1—C8—C7	121.1 (4)
N3—C14—H14A	108.4	N1—C8—H8	119.4
H14B—C14—H14A	107.4	C7—C8—H8	119.4
O5—C4—O4	126.7 (4)	N2—C6—C5	123.1 (4)
O5—C4—C5	118.0 (4)	N2—C6—H6	118.5
O4—C4—C5	115.3 (4)	C5—C6—H6	118.5
C11—C10—N3	115.3 (4)	C10—C11—H11B	109.5
C11—C10—H10A	108.4	C10—C11—H11A	109.5

N3—C10—H10A	108.4	H11B—C11—H11A	109.5
C11—C10—H10B	108.4	C10—C11—H11C	109.5
N3—C10—H10B	108.4	H11B—C11—H11C	109.5
H10A—C10—H10B	107.5	H11A—C11—H11C	109.5
C14—C15—H15B	109.5	O3—C3—Re1	177.0 (4)
C14—C15—H15C	109.5	O2—C2—Re1	177.3 (4)

Fig. 1


O3
C3
O4
O5
O2
C2
Re1
Cl1
C4
C15
C10
C14
C11
C1
N1
C5
N3
C16
O1
C6
C17
C8
C12
C13
N2
C7