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Carbonylchlorido[tris(2-aminoethyl)amine]ruthenium(II) chloride

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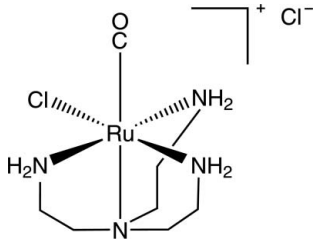
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.003 Å; R factor = 0.020; wR factor = 0.048; data-to-parameter ratio = 20.9.

In the monocationic octahedral complex, [RuCl($C_6H_{18}N_4$)(CO)]Cl, the ruthenium(II) centre is coordinated by a chloride and a carbonyl ligand and by the tetradentate tris(2-aminoethyl)amine (tren) chelator. The complex has approximate non-crystallographic C_s symmetry. In the crystal structure, N—H···Cl hydrogen bonds are found, mainly with the chloride counter-ion as the acceptor.

Related literature

For related literature, see: Sullivan *et al.* (1978); Cremer & Pople (1975).



Experimental

Crystal data

[RuCl($C_6H_{18}N_4$)(CO)]Cl
M_r = 346.22
Monoclinic, $P2_1/c$
a = 10.1514 (2) Å
b = 8.52680 (10) Å
c = 14.7413 (3) Å
β = 101.6790 (7)°
V = 1249.58 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.66 mm^{-1}
T = 200 (2) K
0.17 × 0.12 × 0.07 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.800, T_{max} = 0.890
23413 measured reflections
2863 independent reflections
2562 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.048
S = 1.06
2863 reflections
137 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H41···Cl2^{i}	0.92	2.53	3.3569 (19)	150
N7—H71···Cl2	0.92	2.46	3.2442 (18)	144
N7—H72···Cl2^{i}	0.92	2.36	3.2830 (18)	176
N10—H101···Cl1^{ii}	0.92	2.48	3.3319 (17)	154
N10—H102···Cl2	0.92	2.47	3.3609 (18)	164

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

Data collection: *COLLECT* (Hooft, 1997–2004); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

The authors thank Dr Peter Mayer for professional support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2353).

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Hooft, R. W. W. (1997–2004). *COLLECT*. Bruker–Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2001). *SADABS*. Version 2. University of Göttingen, Germany.
Spek, A. L. (2003). *J Appl. Cryst.* **36**, 7–13.
Sullivan, B. P., Salmon, D. J. & Meyer, T. J. (1978). *Inorg. Chem.* **17**, 3334–3341.

supplementary materials

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Carbonylchlorido[tris(2-aminoethyl)amine]ruthenium(II) chloride

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Comment

The title compound, $C_7H_{18}Cl_2N_4ORu$, was obtained on the attempted preparation of $[RuCl_2(tren)]$ by refluxing $RuCl_3$ with an equimolar amount of tris(2-aminoethyl)amine (tren) in DMF.

The molecular structure is shown in Fig. 1. The complex features three 5-membered rings: Ru–N1–C2–C3–N4 adopts a twist conformation on N1–C2 (Q_2 = 0.444 (2) Å, φ_2 = 241.5 (2)°), whereas Ru–N1–C5–C6–N7 (Q_2 = 0.359 (2) Å, φ_2 = 294.6 (3)°) shows an envelope conformation on C6. Ru–N1–C8–C9–N10 is twisted on N1–C8 (Q_2 = 0.459 (2) Å, φ_2 = 55.6 (2)°). Ring puckering parameters (Cremer & Pople, 1975) were calculated with *PLATON* (Spek, 2007).

Bond lengths and angles are normal.

The molecular packing including the hydrogen bond system is shown in Fig. 2. The search for hydrogen bonded molecular aggregates provided an infinite two-dimensional network (base vectors = [0 1 0], [2 0 1]) along the (1 0 − 2)-plane.

Experimental

The title compound was obtained accidentally on the attempted preparation of $[RuCl_2(tren)]$ by refluxing $RuCl_3 * 3\ H_2O$ with an equimolar amount of tris(2-aminoethyl)amine (tren) and 7 equivalents of LiCl in DMF in analogy to a published procedure (Sullivan *et al.*, 1978).

Refinement

All H atoms were constructed and refined as riding on their parent atoms with one common isotropic displacement parameter.

Figures

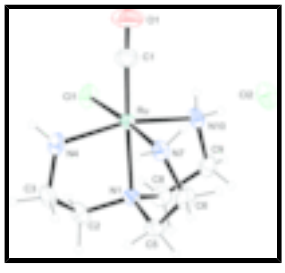

Fig. 1. The molecular structure of (I), with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

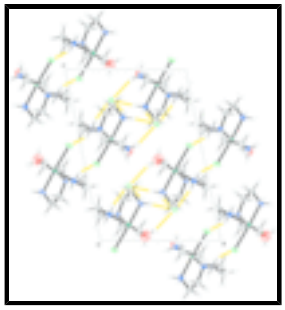

Fig. 2. The packing of (I), viewed along [0 1 0].

Carbonylchlorido[tris(2-aminoethyl)amine]ruthenium(II) chloride

Crystal data

$[RuCl(C_6H_{18}N_4)(CO)]Cl$	$F_{000} = 696$
$M_r = 346.22$	$D_x = 1.840$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 3049 reflections
$a = 10.1514$ (2) Å	$\theta = 3.1$–27.5º
$b = 8.52680$ (10) Å	$\mu = 1.66$ mm^{-1}
$c = 14.7413$ (3) Å	$T = 200$ (2) K
$\beta = 101.6790$ (7)º	Block, yellow
$V = 1249.58$ (4) Å^3	0.17 × 0.12 × 0.07 mm
$Z = 4$	

Data collection

KappaCCD diffractometer	2863 independent reflections
Radiation source: rotating anode	2562 reflections with $I > 2\sigma(I)$
Monochromator: MONTEL, graded multilayered X-ray optics	$R_{int} = 0.031$
$T = 200$(2) K	$\theta_{max} = 27.5$º
φ and ω scans	$\theta_{min} = 3.1$º
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	$h = -13 \rightarrow 13$
$T_{min} = 0.800$, $T_{max} = 0.890$	$k = -11 \rightarrow 10$
23413 measured reflections	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.020$	H-atom parameters constrained
$wR(F^2) = 0.048$	$w = 1/[\sigma^2(F_o^2) + (0.0181P)^2 + 1.1292P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.06	$(\Delta/\sigma)_{max}$ = 0.001
2863 reflections	$\Delta\rho_{max}$ = 0.56 e Å^{-3}
137 parameters	$\Delta\rho_{min}$ = −0.53 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. μ × r = 0.116, T_{min}/T_{max} = 0.899

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

	x	y	z	U_{iso}*/U_{eq}
Ru	0.263803 (15)	0.685462 (18)	0.091163 (10)	0.01895 (6)
Cl1	0.09940 (5)	0.70362 (7)	−0.05312 (3)	0.03170 (13)
O1	0.3830 (2)	0.3964 (2)	0.02630 (15)	0.0593 (5)
N1	0.18612 (16)	0.8875 (2)	0.14884 (11)	0.0220 (3)
N4	0.37625 (18)	0.8578 (2)	0.03603 (12)	0.0285 (4)
H41	0.4667	0.8410	0.0577	0.0381 (16)*
H42	0.3593	0.8499	−0.0275	0.0381 (16)*
N7	0.39797 (17)	0.6894 (2)	0.21966 (12)	0.0247 (4)
H71	0.4162	0.5884	0.2403	0.0381 (16)*
H72	0.4775	0.7352	0.2130	0.0381 (16)*
N10	0.11750 (16)	0.5702 (2)	0.15115 (11)	0.0240 (4)
H101	0.0625	0.5110	0.1068	0.0381 (16)*
H102	0.1595	0.5038	0.1972	0.0381 (16)*
C1	0.3369 (2)	0.5085 (3)	0.04993 (16)	0.0343 (5)
C2	0.1995 (2)	1.0192 (3)	0.08523 (16)	0.0310 (5)
H21	0.1845	1.1202	0.1147	0.0381 (16)*
H22	0.1310	1.0088	0.0273	0.0381 (16)*
C3	0.3396 (2)	1.0170 (3)	0.06317 (16)	0.0329 (5)
H31	0.3428	1.0913	0.0120	0.0381 (16)*
H32	0.4060	1.0521	0.1182	0.0381 (16)*
C5	0.2655 (2)	0.9209 (3)	0.24410 (14)	0.0300 (5)
H51	0.2038	0.9582	0.2836	0.0381 (16)*
H52	0.3308	1.0057	0.2405	0.0381 (16)*
C6	0.3403 (2)	0.7782 (3)	0.28834 (14)	0.0307 (5)
H61	0.4133	0.8114	0.3400	0.0381 (16)*
H62	0.2779	0.7100	0.3141	0.0381 (16)*
C8	0.0435 (2)	0.8471 (3)	0.14896 (16)	0.0299 (5)
H81	−0.0093	0.8482	0.0847	0.0381 (16)*
H82	0.0047	0.9264	0.1851	0.0381 (16)*
C9	0.0346 (2)	0.6870 (3)	0.19081 (15)	0.0292 (5)
H91	0.0668	0.6930	0.2588	0.0381 (16)*
H92	−0.0604	0.6522	0.1785	0.0381 (16)*
Cl2	0.32744 (5)	0.36408 (7)	0.31352 (4)	0.03583 (13)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ru	0.01829 (9)	0.02010 (9)	0.01801 (9)	−0.00241 (6)	0.00263 (6)	−0.00132 (6)
Cl1	0.0317 (3)	0.0424 (3)	0.0181 (2)	−0.0134 (2)	−0.0020 (2)	0.0007 (2)
O1	0.0670 (13)	0.0386 (11)	0.0765 (14)	0.0085 (10)	0.0243 (11)	−0.0213 (10)
N1	0.0230 (8)	0.0200 (8)	0.0226 (8)	0.0004 (7)	0.0039 (7)	0.0014 (7)
N4	0.0253 (9)	0.0384 (11)	0.0225 (8)	−0.0102 (8)	0.0060 (7)	0.0013 (8)
N7	0.0212 (8)	0.0261 (9)	0.0247 (9)	−0.0008 (7)	0.0001 (7)	0.0038 (7)
N10	0.0228 (8)	0.0243 (9)	0.0233 (8)	−0.0079 (7)	0.0007 (7)	0.0025 (7)
C1	0.0345 (12)	0.0319 (12)	0.0371 (12)	−0.0037 (10)	0.0082 (10)	−0.0065 (10)
C2	0.0319 (11)	0.0236 (11)	0.0352 (12)	0.0013 (9)	0.0017 (9)	0.0087 (9)
C3	0.0351 (12)	0.0275 (12)	0.0348 (12)	−0.0104 (9)	0.0045 (9)	0.0082 (10)
C5	0.0385 (12)	0.0267 (11)	0.0239 (10)	−0.0036 (9)	0.0041 (9)	−0.0065 (9)
C6	0.0337 (12)	0.0374 (13)	0.0180 (10)	−0.0053 (10)	−0.0020 (9)	−0.0018 (9)
C8	0.0207 (10)	0.0337 (12)	0.0364 (12)	0.0040 (9)	0.0089 (9)	0.0010 (10)
C9	0.0234 (10)	0.0375 (12)	0.0282 (11)	−0.0055 (9)	0.0087 (8)	0.0010 (9)
Cl2	0.0257 (3)	0.0361 (3)	0.0443 (3)	0.0040 (2)	0.0038 (2)	0.0146 (3)

Geometric parameters (Å, °)

Ru—C1	1.838 (2)	N10—H101	0.9200
Ru—N7	2.0975 (17)	N10—H102	0.9200
Ru—N10	2.1176 (16)	C2—C3	1.522 (3)
Ru—N4	2.1208 (17)	C2—H21	0.9900
Ru—N1	2.1409 (17)	C2—H22	0.9900
Ru—Cl1	2.4281 (5)	C3—H31	0.9900
O1—C1	1.149 (3)	C3—H32	0.9900
N1—C2	1.487 (3)	C5—C6	1.510 (3)
N1—C8	1.489 (3)	C5—H51	0.9900
N1—C5	1.498 (3)	C5—H52	0.9900
N4—C3	1.484 (3)	C6—H61	0.9900
N4—H41	0.9200	C6—H62	0.9900
N4—H42	0.9200	C8—C9	1.508 (3)
N7—C6	1.477 (3)	C8—H81	0.9900
N7—H71	0.9200	C8—H82	0.9900
N7—H72	0.9200	C9—H91	0.9900
N10—C9	1.497 (3)	C9—H92	0.9900
C1—Ru—N7	94.58 (9)	H101—N10—H102	108.1
C1—Ru—N10	96.99 (8)	O1—C1—Ru	178.3 (2)
N7—Ru—N10	90.72 (6)	N1—C2—C3	109.53 (17)
C1—Ru—N4	99.04 (9)	N1—C2—H21	109.8
N7—Ru—N4	91.91 (7)	C3—C2—H21	109.8
N10—Ru—N4	163.50 (7)	N1—C2—H22	109.8
C1—Ru—N1	175.95 (9)	C3—C2—H22	109.8
N7—Ru—N1	81.51 (6)	H21—C2—H22	108.2
N10—Ru—N1	82.04 (6)	N4—C3—C2	111.40 (17)

N4—Ru—N1	82.24 (7)	N4—C3—H31	109.3
C1—Ru—Cl1	90.85 (7)	C2—C3—H31	109.3
N7—Ru—Cl1	174.57 (5)	N4—C3—H32	109.3
N10—Ru—Cl1	88.83 (5)	C2—C3—H32	109.3
N4—Ru—Cl1	87.02 (5)	H31—C3—H32	108.0
N1—Ru—Cl1	93.06 (5)	N1—C5—C6	112.32 (17)
C2—N1—C8	112.88 (16)	N1—C5—H51	109.1
C2—N1—C5	110.32 (16)	C6—C5—H51	109.1
C8—N1—C5	112.42 (16)	N1—C5—H52	109.1
C2—N1—Ru	105.61 (12)	C6—C5—H52	109.1
C8—N1—Ru	104.53 (12)	H51—C5—H52	107.9
C5—N1—Ru	110.72 (12)	N7—C6—C5	110.56 (17)
C3—N4—Ru	110.29 (12)	N7—C6—H61	109.5
C3—N4—H41	109.6	C5—C6—H61	109.5
Ru—N4—H41	109.6	N7—C6—H62	109.5
C3—N4—H42	109.6	C5—C6—H62	109.5
Ru—N4—H42	109.6	H61—C6—H62	108.1
H41—N4—H42	108.1	N1—C8—C9	110.39 (17)
C6—N7—Ru	110.62 (12)	N1—C8—H81	109.6
C6—N7—H71	109.5	C9—C8—H81	109.6
Ru—N7—H71	109.5	N1—C8—H82	109.6
C6—N7—H72	109.5	C9—C8—H82	109.6
Ru—N7—H72	109.5	H81—C8—H82	108.1
H71—N7—H72	108.1	N10—C9—C8	110.95 (16)
C9—N10—Ru	110.59 (12)	N10—C9—H91	109.4
C9—N10—H101	109.5	C8—C9—H91	109.4
Ru—N10—H101	109.5	N10—C9—H92	109.4
C9—N10—H102	109.5	C8—C9—H92	109.4
Ru—N10—H102	109.5	H91—C9—H92	108.0
N7—Ru—N1—C2	119.66 (13)	C1—Ru—N10—C9	−178.62 (14)
N10—Ru—N1—C2	−148.41 (13)	N7—Ru—N10—C9	86.67 (13)
N4—Ru—N1—C2	26.54 (12)	N4—Ru—N10—C9	−12.5 (3)
Cl1—Ru—N1—C2	−60.03 (12)	N1—Ru—N10—C9	5.35 (13)
N7—Ru—N1—C8	−121.04 (13)	Cl1—Ru—N10—C9	−87.91 (12)
N10—Ru—N1—C8	−29.11 (12)	C8—N1—C2—C3	−160.53 (18)
N4—Ru—N1—C8	145.84 (13)	C5—N1—C2—C3	72.8 (2)
Cl1—Ru—N1—C8	59.28 (12)	Ru—N1—C2—C3	−46.91 (19)
N7—Ru—N1—C5	0.25 (13)	Ru—N4—C3—C2	−23.8 (2)
N10—Ru—N1—C5	92.17 (13)	N1—C2—C3—N4	48.3 (2)
N4—Ru—N1—C5	−92.87 (13)	C2—N1—C5—C6	−138.18 (19)
Cl1—Ru—N1—C5	−179.44 (12)	C8—N1—C5—C6	94.9 (2)
C1—Ru—N4—C3	−177.75 (15)	Ru—N1—C5—C6	−21.6 (2)
N7—Ru—N4—C3	−82.80 (14)	Ru—N7—C6—C5	−39.3 (2)
N10—Ru—N4—C3	16.2 (3)	N1—C5—C6—N7	40.3 (2)
N1—Ru—N4—C3	−1.64 (13)	C2—N1—C8—C9	163.23 (18)
Cl1—Ru—N4—C3	91.87 (13)	C5—N1—C8—C9	−71.2 (2)
C1—Ru—N7—C6	−157.49 (15)	Ru—N1—C8—C9	48.95 (19)
N10—Ru—N7—C6	−60.42 (14)	Ru—N10—C9—C8	20.0 (2)
N4—Ru—N7—C6	103.28 (14)	N1—C8—C9—N10	−47.2 (2)

N1—Ru—N7—C6	21.42 (13)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H41···Cl2^{i}	0.92	2.53	3.3569 (19)	150
N7—H71···Cl2	0.92	2.46	3.2442 (18)	144
N7—H72···Cl2^{i}	0.92	2.36	3.2830 (18)	176
N10—H101···Cl1^{ii}	0.92	2.48	3.3319 (17)	154
N10—H102···Cl2	0.92	2.47	3.3609 (18)	164

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

Fig. 1

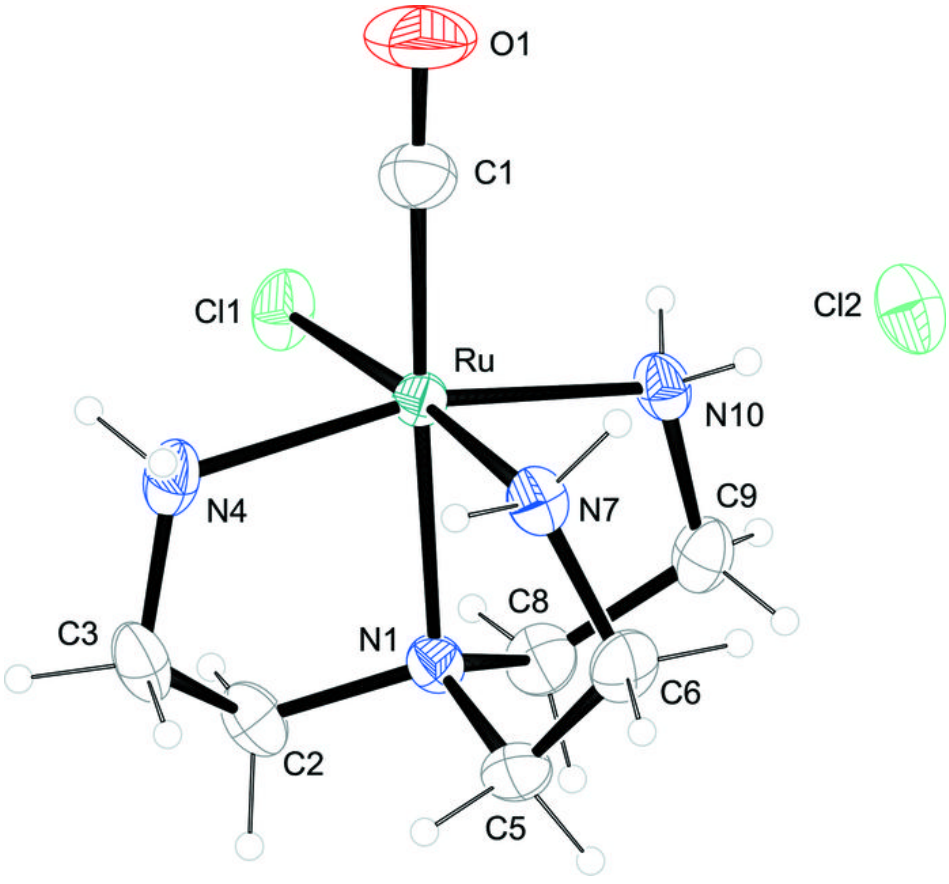

Fig. 2