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(Dibenzo-18-crown-6)(2-phenylamidopyridine)-rubidium(I)

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Key indicators

Single-crystal X-ray study
T = 160 K
Mean σ(C–C) = 0.003 Å
R factor = 0.027
wR factor = 0.062
Data-to-parameter ratio = 14.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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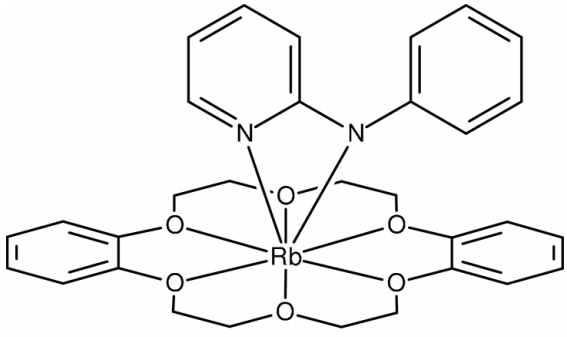
The title complex, $[Rb(C_{11}H_9N_2)(C_{20}H_{24}O_6)]$, has rubidium in an irregular eightfold coordination, with a hexadentate crown ether ligand and a chelating bidentate amidopyridine ligand, each occupying one hemisphere of the coordination. The chelate RbNCN ring is essentially planar, but the two rings of the amidopyridine ligand are not coplanar, because of steric interaction of H atoms on the rings. The Rb—N(amido) bond is shorter than the Rb—N(pyridine) bond.

Comment

We have recently reported the synthesis and structural characterization of a range of complexes of alkali metals (Li—Cs) with the 2-phenylamidopyridine ligand (L^-) and the crown ethers 12-crown-4, 15-crown-5 and 18-crown-6 (Liddle & Clegg, 2003; Liddle *et al.*, 2004). This included the complex (18-crown-6)Rb(L). We have also prepared the related complex with dibenzo-18-crown-6, and report its structure here.



(I)

Like the analogous complex (18-crown-6)Rb(L), the title complex (dibenzo-18-crown-6)Rb(L), (I), consists of discrete neutral molecules with no special intermolecular interactions. The crown ligand is hexadentate and occupies one coordination hemisphere, with the bidentate amide ligand occupying the other, giving irregular eightfold coordination for the rubidium ion. The complex may be described as a contact ion-pair, in contrast to several of the crown-ligated complexes of alkali metals with the amide L^-, which exist in the solid state as separated ion pairs (Liddle *et al.*, 2004).

The molecular structure of (I) (Fig. 1 and Table 1) is very similar to that of the (18-crown-6) complex, which has already been described and compared with related complexes (Liddle *et al.*, 2004). The main differences are probably due predominantly to the greater rigidity of dibenzo-18-crown-6 compared with 18-crown-6. The range of Rb—O bond lengths is somewhat smaller, and the two Rb—N bonds are both

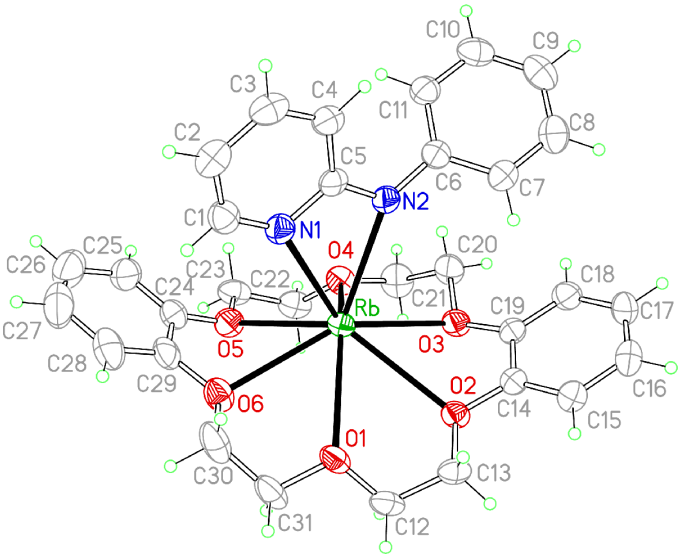


Figure 1
The molecular structure of (I), showing the atom labels and 50% probability displacement ellipsoids for non-H atoms.

shorter in (I), by about 0.05–0.08 Å, with the shorter bond to the amide N atom in both complexes. The Rb^+ ion lies 1.0945 (6) Å out of the mean plane of the six O atoms of the crown (r.m.s. deviation 0.111 Å), displaced towards the amide ligand, and the two benzene rings of the crown are folded out of the oxygen mean plane towards the amide ligand by 22.93 (7) and 25.94 (7)°, presumably as a result of intermolecular packing interactions in the absence of an obvious electronic factor. As expected, the crown ether ring displays a sequence of *anti* (or *trans*) conformations about C—C—O—C and *gauche* conformations about O—C—C—O linkages, except for the near-zero torsion angles for O—C—C—O at the constrained benzo fusions (Table 1).

Chelation by the amide ligand forces a *syn* arrangement for the two N atoms. The two rings are not coplanar, however, because of steric interaction of the H atoms bonded to C4 and C11; the dihedral angle between the rings is 58.10 (5)°. The four-membered chelate ring (RbNCN) is essentially planar, the dihedral angle between the RbN_2 and CN_2 planes being only 0.3 (3)°.

Experimental

n-Butyllithium (0.4 ml of a 2.5 *M* solution in hexanes, 1.0 mmol) was added dropwise to a solution of 2-phenylaminopyridine (0.17 g, 1.0 mmol) and dibenzo-18-crown-6 (0.36 g, 1.0 mmol) in tetrahydrofuran (THF, 40 ml), followed by rubidium 2-ethylhexoxide (0.22 g, 1.0 mmol) in THF (10 ml), to give a pale yellow precipitate. Volatile components were removed *in vacuo* and the remaining solid was washed with petroleum ether (3 × 5 ml). Recrystallization from hot toluene containing a little hexamethylphosphoramide (HMPA) gave yellow crystals of (I) (yield 0.31 g, 51%). Chemical analysis results were satisfactory, and the ^{1}H and ^{13}C{^{1}H} NMR signals could be assigned on the basis of the crystal structure (Liddle, 2000).

Crystal data

$[Rb(C_{11}H_9N_2)(C_{20}H_{24}O_6)]$
M_r = 615.06
Monoclinic, $P2_1/n$
a = 8.1464 (5) Å
b = 23.5768 (14) Å
c = 15.2385 (9) Å
β = 96.626 (2)°
V = 2907.2 (3) Å^3
Z = 4
D_x = 1.405 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 14292 reflections
θ = 2.7–28.3°
μ = 1.75 mm^{-1}
T = 160 (2) K
Plate, yellow
0.88 × 0.62 × 0.10 mm

Data collection

Bruker SMART 1K CCD diffractometer
Thin-slice ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.308, T_{max} = 0.845
20580 measured reflections
5112 independent reflections
3890 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 25.0°
$h = -9 \rightarrow 9$
$k = -28 \rightarrow 28$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.062
S = 0.92
5112 reflections
361 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0307P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.62 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Rb—N1	2.9441 (18)	Rb—O5	2.9593 (14)
Rb—N2	2.9025 (17)	Rb—O6	2.8829 (15)
Rb—O1	2.9640 (14)	N1—C1	1.331 (3)
Rb—O2	2.9700 (14)	N1—C5	1.378 (3)
Rb—O3	2.9536 (15)	N2—C5	1.332 (3)
Rb—O4	2.9508 (14)	N2—C6	1.385 (3)
N1—Rb—N2	46.13 (5)	Rb—N1—C1	143.59 (15)
O1—Rb—O2	57.05 (4)	Rb—N1—C5	97.67 (13)
O1—Rb—O6	57.59 (4)	C1—N1—C5	118.6 (2)
O2—Rb—O3	51.74 (4)	Rb—N2—C5	100.80 (13)
O3—Rb—O4	58.15 (4)	Rb—N2—C6	136.58 (14)
O4—Rb—O5	56.03 (4)	C5—N2—C6	121.37 (18)
O5—Rb—O6	51.94 (4)	N1—C5—N2	115.39 (19)
C6—N2—C5—N1	169.51 (18)	C22—O4—C21—C20	−166.07 (18)
C6—N2—C5—C4	−14.6 (3)	O3—C20—C21—O4	68.4 (2)
C5—N2—C6—C7	136.0 (2)	C21—O4—C22—C23	−168.59 (17)
C5—N2—C6—C11	−50.3 (3)	C24—O5—C23—C22	176.63 (18)
C31—O1—C12—C13	−174.83 (17)	O4—C22—C23—O5	−61.3 (2)
C14—O2—C13—C12	−176.31 (17)	C23—O5—C24—C29	173.54 (18)
O1—C12—C13—O2	−64.4 (2)	C30—O6—C29—C24	−176.59 (19)
C13—O2—C14—C19	178.36 (17)	O5—C24—C29—O6	−0.9 (3)
C20—O3—C19—C14	169.43 (19)	C29—O6—C30—C31	167.46 (19)
O2—C14—C19—O3	−1.4 (3)	C12—O1—C31—C30	−173.43 (18)
C19—O3—C20—C21	164.94 (19)	O6—C30—C31—O1	65.1 (2)

H atoms were positioned geometrically, with C—H = 0.95 (aromatic) or 0.99 Å (aliphatic), and refined with a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 2001); cell refinement: local programs; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

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supporting information

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Crystal data

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$V = 2907.2$ (3) Å^3
$Z = 4$
$F(000) = 1272$
$D_x = 1.405$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 14292 reflections
$\theta = 2.7$–$28.3°$
$\mu = 1.75$ mm^{-1}
$T = 160$ K
Plate, yellow
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Graphite monochromator
Detector resolution: 8.192 pixels mm^{-1}
thin–slice ω scans
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5112 independent reflections
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$R_{int} = 0.050$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.2°$
$h = -9 \rightarrow 9$
$k = -28 \rightarrow 28$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.062$
$S = 0.92$
5112 reflections
361 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
$w = 1/[\sigma^2(F_o^2) + (0.0307P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.45$ e Å^{-3}
$\Delta\rho_{min} = -0.62$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Rb	0.52699 (2)	0.235217 (9)	0.613068 (13)	0.02988 (7)
N1	0.6494 (2)	0.30274 (8)	0.47167 (12)	0.0372 (4)
N2	0.6620 (2)	0.20672 (8)	0.45054 (11)	0.0375 (4)

C1	0.6824 (3)	0.35533 (10)	0.44727 (15)	0.0411 (6)
H1A	0.6520	0.3854	0.4837	0.049*
C2	0.7572 (3)	0.37007 (11)	0.37349 (16)	0.0440 (6)
H2A	0.7782	0.4085	0.3596	0.053*
C3	0.7997 (2)	0.32559 (11)	0.32086 (15)	0.0428 (6)
H3A	0.8525	0.3334	0.2698	0.051*
C4	0.7668 (2)	0.27080 (10)	0.34160 (14)	0.0369 (5)
H4A	0.7946	0.2408	0.3043	0.044*
C5	0.6901 (2)	0.25826 (10)	0.41967 (14)	0.0331 (5)
C6	0.6719 (2)	0.15876 (9)	0.39893 (14)	0.0341 (5)
C7	0.7533 (3)	0.11077 (9)	0.43663 (15)	0.0368 (5)
H7A	0.8055	0.1127	0.4956	0.044*
C8	0.7595 (3)	0.06048 (10)	0.39012 (17)	0.0467 (6)
H8A	0.8194	0.0292	0.4168	0.056*
C9	0.6800 (3)	0.05528 (11)	0.30566 (17)	0.0487 (6)
H9A	0.6833	0.0206	0.2742	0.058*
C10	0.5955 (3)	0.10156 (11)	0.26756 (16)	0.0470 (6)
H10A	0.5387	0.0984	0.2097	0.056*
C11	0.5926 (3)	0.15212 (10)	0.31246 (14)	0.0412 (6)
H11A	0.5354	0.1835	0.2843	0.049*
O1	0.71259 (17)	0.27684 (6)	0.77934 (9)	0.0371 (4)
O2	0.69327 (16)	0.15823 (6)	0.74983 (9)	0.0330 (3)
O3	0.46764 (17)	0.11303 (6)	0.63832 (9)	0.0372 (4)
O4	0.19887 (16)	0.18357 (6)	0.56543 (10)	0.0392 (4)
O5	0.21222 (17)	0.29886 (7)	0.60252 (10)	0.0405 (4)
O6	0.46830 (17)	0.34525 (6)	0.68588 (10)	0.0430 (4)
C12	0.7421 (3)	0.23615 (10)	0.84822 (14)	0.0389 (6)
H12A	0.6369	0.2271	0.8718	0.047*
H12B	0.8191	0.2522	0.8969	0.047*
C13	0.8142 (2)	0.18332 (9)	0.81480 (13)	0.0352 (5)
H13A	0.9160	0.1924	0.7879	0.042*
H13B	0.8431	0.1565	0.8641	0.042*
C14	0.7337 (2)	0.10703 (9)	0.71488 (13)	0.0301 (5)
C15	0.8822 (3)	0.07934 (9)	0.73676 (15)	0.0375 (5)
H15A	0.9636	0.0952	0.7794	0.045*
C16	0.9120 (3)	0.02810 (10)	0.69613 (16)	0.0440 (6)
H16A	1.0146	0.0093	0.7105	0.053*
C17	0.7946 (3)	0.00479 (10)	0.63580 (16)	0.0462 (6)
H17A	0.8162	−0.0301	0.6082	0.055*
C18	0.6441 (3)	0.03162 (9)	0.61451 (15)	0.0413 (6)
H18A	0.5624	0.0148	0.5730	0.050*
C19	0.6123 (3)	0.08280 (9)	0.65361 (13)	0.0313 (5)
C20	0.3492 (3)	0.09615 (10)	0.56578 (16)	0.0445 (6)
H20A	0.3360	0.0544	0.5652	0.053*
H20B	0.3870	0.1080	0.5091	0.053*
C21	0.1891 (3)	0.12404 (11)	0.57746 (18)	0.0499 (7)
H21A	0.1009	0.1083	0.5342	0.060*
H21B	0.1599	0.1160	0.6375	0.060*

C22	0.0628 (3)	0.21261 (11)	0.59646 (15)	0.0433 (6)
H22A	0.0732	0.2114	0.6618	0.052*
H22B	−0.0421	0.1938	0.5735	0.052*
C23	0.0613 (3)	0.27241 (10)	0.56595 (15)	0.0434 (6)
H23A	0.0517	0.2738	0.5006	0.052*
H23B	−0.0344	0.2927	0.5856	0.052*
C24	0.2397 (3)	0.35388 (10)	0.57851 (15)	0.0413 (6)
C25	0.1401 (3)	0.38451 (12)	0.51689 (16)	0.0546 (7)
H25A	0.0436	0.3677	0.4868	0.066*
C26	0.1803 (4)	0.43995 (13)	0.4987 (2)	0.0704 (9)
H26A	0.1114	0.4610	0.4559	0.085*
C27	0.3186 (4)	0.46476 (12)	0.5417 (2)	0.0709 (9)
H27A	0.3450	0.5029	0.5287	0.085*
C28	0.4210 (3)	0.43433 (11)	0.60471 (18)	0.0565 (7)
H28A	0.5175	0.4515	0.6342	0.068*
C29	0.3816 (3)	0.37929 (10)	0.62396 (16)	0.0420 (6)
C30	0.6087 (3)	0.36889 (10)	0.73831 (18)	0.0548 (7)
H30A	0.5804	0.4064	0.7616	0.066*
H30B	0.7010	0.3739	0.7021	0.066*
C31	0.6576 (3)	0.32908 (10)	0.81257 (17)	0.0495 (7)
H31A	0.7473	0.3462	0.8535	0.059*
H31B	0.5621	0.3221	0.8458	0.059*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Rb	0.03005 (11)	0.03301 (12)	0.02705 (11)	−0.00264 (9)	0.00528 (8)	−0.00251 (10)
N1	0.0386 (10)	0.0382 (12)	0.0357 (10)	0.0045 (9)	0.0077 (8)	0.0035 (9)
N2	0.0458 (11)	0.0376 (12)	0.0307 (10)	0.0096 (9)	0.0117 (8)	0.0058 (9)
C1	0.0376 (13)	0.0418 (15)	0.0437 (14)	0.0031 (11)	0.0034 (11)	−0.0014 (12)
C2	0.0351 (13)	0.0475 (16)	0.0488 (15)	−0.0038 (11)	0.0017 (11)	0.0110 (12)
C3	0.0281 (12)	0.0649 (18)	0.0364 (13)	0.0013 (11)	0.0078 (10)	0.0097 (13)
C4	0.0272 (11)	0.0512 (16)	0.0334 (12)	0.0087 (11)	0.0081 (9)	0.0051 (11)
C5	0.0235 (10)	0.0456 (14)	0.0296 (11)	0.0099 (10)	0.0006 (9)	0.0034 (11)
C6	0.0284 (11)	0.0413 (14)	0.0350 (13)	0.0037 (10)	0.0140 (10)	0.0044 (11)
C7	0.0313 (12)	0.0405 (14)	0.0385 (13)	−0.0008 (10)	0.0036 (10)	0.0045 (11)
C8	0.0403 (14)	0.0379 (15)	0.0625 (17)	−0.0011 (11)	0.0085 (12)	0.0017 (13)
C9	0.0471 (15)	0.0431 (16)	0.0584 (17)	−0.0071 (12)	0.0171 (13)	−0.0125 (13)
C10	0.0405 (14)	0.0644 (18)	0.0379 (14)	−0.0029 (13)	0.0121 (11)	−0.0050 (13)
C11	0.0391 (13)	0.0502 (16)	0.0353 (13)	0.0093 (11)	0.0086 (10)	0.0035 (12)
O1	0.0356 (8)	0.0378 (9)	0.0371 (9)	0.0011 (7)	0.0009 (7)	−0.0139 (7)
O2	0.0270 (7)	0.0362 (9)	0.0347 (8)	−0.0011 (6)	−0.0017 (6)	−0.0076 (7)
O3	0.0323 (8)	0.0400 (9)	0.0373 (9)	−0.0002 (7)	−0.0045 (7)	−0.0074 (7)
O4	0.0294 (8)	0.0442 (10)	0.0438 (9)	−0.0014 (7)	0.0037 (7)	−0.0069 (7)
O5	0.0331 (8)	0.0454 (10)	0.0412 (9)	0.0030 (7)	−0.0030 (7)	−0.0030 (8)
O6	0.0366 (9)	0.0385 (10)	0.0528 (10)	0.0010 (7)	0.0004 (8)	−0.0095 (8)
C12	0.0325 (12)	0.0541 (15)	0.0294 (12)	−0.0017 (11)	0.0001 (10)	−0.0127 (12)
C13	0.0276 (11)	0.0474 (15)	0.0295 (12)	−0.0051 (10)	−0.0013 (9)	−0.0032 (10)

C14	0.0314 (12)	0.0286 (12)	0.0311 (12)	−0.0038 (9)	0.0070 (9)	0.0030 (10)
C15	0.0337 (12)	0.0369 (14)	0.0413 (13)	−0.0021 (10)	0.0019 (10)	0.0020 (11)
C16	0.0419 (14)	0.0358 (14)	0.0547 (16)	0.0075 (11)	0.0076 (12)	0.0073 (12)
C17	0.0610 (16)	0.0292 (13)	0.0484 (15)	0.0066 (12)	0.0069 (13)	0.0012 (11)
C18	0.0511 (15)	0.0333 (14)	0.0382 (13)	−0.0053 (11)	−0.0012 (11)	−0.0022 (11)
C19	0.0326 (12)	0.0315 (13)	0.0303 (12)	−0.0021 (10)	0.0054 (10)	0.0039 (10)
C20	0.0421 (14)	0.0375 (14)	0.0501 (15)	−0.0057 (11)	−0.0100 (12)	−0.0098 (12)
C21	0.0337 (13)	0.0485 (17)	0.0647 (18)	−0.0145 (11)	−0.0069 (12)	−0.0076 (13)
C22	0.0279 (12)	0.0597 (17)	0.0416 (14)	−0.0052 (11)	0.0007 (10)	−0.0077 (12)
C23	0.0285 (12)	0.0656 (19)	0.0347 (13)	0.0058 (11)	−0.0015 (10)	−0.0107 (12)
C24	0.0415 (13)	0.0430 (15)	0.0413 (14)	0.0141 (12)	0.0128 (11)	−0.0039 (12)
C25	0.0584 (17)	0.0576 (18)	0.0479 (16)	0.0201 (14)	0.0063 (13)	0.0017 (14)
C26	0.080 (2)	0.067 (2)	0.065 (2)	0.0294 (18)	0.0117 (17)	0.0155 (17)
C27	0.088 (2)	0.0448 (18)	0.086 (2)	0.0196 (17)	0.0361 (19)	0.0135 (16)
C28	0.0555 (17)	0.0435 (16)	0.074 (2)	0.0072 (13)	0.0237 (15)	−0.0057 (15)
C29	0.0444 (14)	0.0369 (14)	0.0469 (15)	0.0107 (11)	0.0146 (12)	−0.0065 (12)
C30	0.0447 (15)	0.0355 (15)	0.082 (2)	−0.0017 (12)	−0.0028 (14)	−0.0230 (14)
C31	0.0413 (14)	0.0450 (15)	0.0593 (17)	0.0045 (12)	−0.0067 (12)	−0.0288 (13)

Geometric parameters (Å, º)

Rb—N1	2.9441 (18)	O6—C30	1.430 (3)
Rb—N2	2.9025 (17)	C12—H12A	0.990
Rb—O1	2.9640 (14)	C12—H12B	0.990
Rb—O2	2.9700 (14)	C12—C13	1.492 (3)
Rb—O3	2.9536 (15)	C13—H13A	0.990
Rb—O4	2.9508 (14)	C13—H13B	0.990
Rb—O5	2.9593 (14)	C14—C15	1.381 (3)
Rb—O6	2.8829 (15)	C14—C19	1.401 (3)
N1—C1	1.331 (3)	C15—H15A	0.950
N1—C5	1.378 (3)	C15—C16	1.392 (3)
N2—C5	1.332 (3)	C16—H16A	0.950
N2—C6	1.385 (3)	C16—C17	1.363 (3)
C1—H1A	0.950	C17—H17A	0.950
C1—C2	1.383 (3)	C17—C18	1.385 (3)
C2—H2A	0.950	C18—H18A	0.950
C2—C3	1.388 (3)	C18—C19	1.383 (3)
C3—H3A	0.950	C20—H20A	0.990
C3—C4	1.364 (3)	C20—H20B	0.990
C4—H4A	0.950	C20—C21	1.489 (3)
C4—C5	1.437 (3)	C21—H21A	0.990
C6—C7	1.401 (3)	C21—H21B	0.990
C6—C11	1.408 (3)	C22—H22A	0.990
C7—H7A	0.950	C22—H22B	0.990
C7—C8	1.385 (3)	C22—C23	1.484 (3)
C8—H8A	0.950	C23—H23A	0.990
C8—C9	1.378 (3)	C23—H23B	0.990
C9—H9A	0.950	C24—C25	1.373 (3)

C9—C10	1.382 (3)	C24—C29	1.411 (3)
C10—H10A	0.950	C25—H25A	0.950
C10—C11	1.376 (3)	C25—C26	1.384 (4)
C11—H11A	0.950	C26—H26A	0.950
O1—C12	1.422 (3)	C26—C27	1.368 (4)
O1—C31	1.424 (2)	C27—H27A	0.950
O2—C13	1.441 (2)	C27—C28	1.395 (4)
O2—C14	1.375 (2)	C28—H28A	0.950
O3—C19	1.374 (2)	C28—C29	1.376 (3)
O3—C20	1.437 (2)	C30—H30A	0.990
O4—C21	1.419 (3)	C30—H30B	0.990
O4—C22	1.429 (3)	C30—C31	1.489 (4)
O5—C23	1.433 (2)	C31—H31A	0.990
O5—C24	1.373 (3)	C31—H31B	0.990
O6—C29	1.371 (3)		

N1—Rb—N2	46.13 (5)	O1—C12—H12A	109.5
N1—Rb—O1	105.32 (5)	O1—C12—H12B	109.5
N1—Rb—O2	132.72 (4)	O1—C12—C13	110.55 (17)
N1—Rb—O3	134.20 (5)	H12A—C12—H12B	108.1
N1—Rb—O4	114.63 (5)	H12A—C12—C13	109.5
N1—Rb—O5	92.90 (5)	H12B—C12—C13	109.5
N1—Rb—O6	83.13 (5)	O2—C13—C12	108.20 (16)
N2—Rb—O1	126.87 (5)	O2—C13—H13A	110.1
N2—Rb—O2	105.67 (4)	O2—C13—H13B	110.1
N2—Rb—O3	88.23 (5)	C12—C13—H13A	110.1
N2—Rb—O4	96.68 (5)	C12—C13—H13B	110.1
N2—Rb—O5	118.84 (4)	H13A—C13—H13B	108.4
N2—Rb—O6	129.25 (5)	O2—C14—C15	124.32 (18)
O1—Rb—O2	57.05 (4)	O2—C14—C19	115.71 (17)
O1—Rb—O3	106.65 (4)	C15—C14—C19	120.0 (2)
O1—Rb—O4	134.85 (4)	C14—C15—H15A	120.1
O1—Rb—O5	103.41 (4)	C14—C15—C16	119.7 (2)
O1—Rb—O6	57.59 (4)	H15A—C15—C16	120.1
O2—Rb—O3	51.74 (4)	C15—C16—H16A	119.8
O2—Rb—O4	104.32 (4)	C15—C16—C17	120.3 (2)
O2—Rb—O5	132.08 (4)	H16A—C16—C17	119.8
O2—Rb—O6	111.24 (4)	C16—C17—H17A	119.8
O3—Rb—O4	58.15 (4)	C16—C17—C18	120.5 (2)
O3—Rb—O5	110.35 (4)	H17A—C17—C18	119.8
O3—Rb—O6	142.24 (4)	C17—C18—H18A	119.9
O4—Rb—O5	56.03 (4)	C17—C18—C19	120.1 (2)
O4—Rb—O6	106.28 (4)	H18A—C18—C19	119.9
O5—Rb—O6	51.94 (4)	O3—C19—C14	115.30 (18)
Rb—N1—C1	143.59 (15)	O3—C19—C18	125.37 (19)
Rb—N1—C5	97.67 (13)	C14—C19—C18	119.33 (19)
C1—N1—C5	118.6 (2)	O3—C20—H20A	110.2
Rb—N2—C5	100.80 (13)	O3—C20—H20B	110.2

Rb—N2—C6	136.58 (14)	O3—C20—C21	107.77 (19)
C5—N2—C6	121.37 (18)	H20A—C20—H20B	108.5
N1—C1—H1A	117.2	H20A—C20—C21	110.2
N1—C1—C2	125.7 (2)	H20B—C20—C21	110.2
H1A—C1—C2	117.2	O4—C21—C20	111.02 (18)
C1—C2—H2A	121.9	O4—C21—H21A	109.4
C1—C2—C3	116.3 (2)	O4—C21—H21B	109.4
H2A—C2—C3	121.9	C20—C21—H21A	109.4
C2—C3—H3A	119.6	C20—C21—H21B	109.4
C2—C3—C4	120.8 (2)	H21A—C21—H21B	108.0
H3A—C3—C4	119.6	O4—C22—H22A	109.8
C3—C4—H4A	119.9	O4—C22—H22B	109.8
C3—C4—C5	120.2 (2)	O4—C22—C23	109.26 (18)
H4A—C4—C5	119.9	H22A—C22—H22B	108.3
N1—C5—N2	115.39 (19)	H22A—C22—C23	109.8
N1—C5—C4	118.4 (2)	H22B—C22—C23	109.8
N2—C5—C4	126.1 (2)	O5—C23—C22	108.42 (17)
N2—C6—C7	118.8 (2)	O5—C23—H23A	110.0
N2—C6—C11	124.9 (2)	O5—C23—H23B	110.0
C7—C6—C11	116.0 (2)	C22—C23—H23A	110.0
C6—C7—H7A	119.2	C22—C23—H23B	110.0
C6—C7—C8	121.7 (2)	H23A—C23—H23B	108.4
H7A—C7—C8	119.2	O5—C24—C25	125.3 (2)
C7—C8—H8A	119.6	O5—C24—C29	114.8 (2)
C7—C8—C9	120.9 (2)	C25—C24—C29	119.9 (2)
H8A—C8—C9	119.6	C24—C25—H25A	120.0
C8—C9—H9A	120.7	C24—C25—C26	119.9 (3)
C8—C9—C10	118.7 (2)	H25A—C25—C26	120.0
H9A—C9—C10	120.7	C25—C26—H26A	119.7
C9—C10—H10A	119.6	C25—C26—C27	120.6 (3)
C9—C10—C11	120.8 (2)	H26A—C26—C27	119.7
H10A—C10—C11	119.6	C26—C27—H27A	119.9
C6—C11—C10	122.0 (2)	C26—C27—C28	120.3 (3)
C6—C11—H11A	119.0	H27A—C27—C28	119.9
C10—C11—H11A	119.0	C27—C28—H28A	120.1
Rb—O1—C12	115.71 (11)	C27—C28—C29	119.7 (3)
Rb—O1—C31	116.12 (12)	H28A—C28—C29	120.1
C12—O1—C31	110.76 (17)	O6—C29—C24	114.7 (2)
Rb—O2—C13	116.85 (12)	O6—C29—C28	125.8 (2)
Rb—O2—C14	112.03 (11)	C24—C29—C28	119.6 (2)
C13—O2—C14	116.82 (15)	O6—C30—H30A	110.1
Rb—O3—C19	112.20 (11)	O6—C30—H30B	110.1
Rb—O3—C20	105.98 (12)	O6—C30—C31	107.8 (2)
C19—O3—C20	118.26 (16)	H30A—C30—H30B	108.5
Rb—O4—C21	116.04 (11)	H30A—C30—C31	110.1
Rb—O4—C22	115.86 (12)	H30B—C30—C31	110.1
C21—O4—C22	111.97 (17)	O1—C31—C30	110.0 (2)
Rb—O5—C23	119.98 (13)	O1—C31—H31A	109.7

Rb—O5—C24	109.06 (12)	O1—C31—H31B	109.7
C23—O5—C24	117.81 (17)	C30—C31—H31A	109.7
Rb—O6—C29	110.92 (12)	C30—C31—H31B	109.7
Rb—O6—C30	114.10 (12)	H31A—C31—H31B	108.2
C29—O6—C30	118.13 (18)		

N2—Rb—N1—C1	−175.0 (3)	N2—Rb—O4—C21	81.14 (15)
N2—Rb—N1—C5	0.18 (10)	N2—Rb—O4—C22	−144.48 (14)
O1—Rb—N1—C1	−48.2 (2)	O1—Rb—O4—C21	−84.42 (15)
O1—Rb—N1—C5	127.02 (11)	O1—Rb—O4—C22	49.96 (16)
O2—Rb—N1—C1	−107.2 (2)	O2—Rb—O4—C21	−26.96 (15)
O2—Rb—N1—C5	67.99 (13)	O2—Rb—O4—C22	107.43 (14)
O3—Rb—N1—C1	178.9 (2)	O3—Rb—O4—C21	−2.59 (14)
O3—Rb—N1—C5	−5.89 (14)	O3—Rb—O4—C22	131.80 (15)
O4—Rb—N1—C1	110.3 (2)	O5—Rb—O4—C21	−158.41 (16)
O4—Rb—N1—C5	−74.55 (12)	O5—Rb—O4—C22	−24.03 (13)
O5—Rb—N1—C1	56.6 (2)	O6—Rb—O4—C21	−144.61 (14)
O5—Rb—N1—C5	−128.23 (11)	O6—Rb—O4—C22	−10.22 (14)
O6—Rb—N1—C1	5.5 (2)	N1—Rb—O5—C23	108.41 (15)
O6—Rb—N1—C5	−179.34 (12)	N1—Rb—O5—C24	−31.86 (13)
N1—Rb—N2—C5	−0.19 (11)	N2—Rb—O5—C23	68.26 (15)
N1—Rb—N2—C6	−166.7 (2)	N2—Rb—O5—C24	−72.01 (14)
O1—Rb—N2—C5	−74.95 (13)	O1—Rb—O5—C23	−145.09 (14)
O1—Rb—N2—C6	118.52 (19)	O1—Rb—O5—C24	74.64 (13)
O2—Rb—N2—C5	−135.24 (11)	O2—Rb—O5—C23	−87.64 (15)
O2—Rb—N2—C6	58.2 (2)	O2—Rb—O5—C24	132.09 (12)
O3—Rb—N2—C5	175.46 (12)	O3—Rb—O5—C23	−31.34 (15)
O3—Rb—N2—C6	8.93 (19)	O3—Rb—O5—C24	−171.61 (12)
O4—Rb—N2—C5	117.82 (12)	O4—Rb—O5—C23	−9.56 (14)
O4—Rb—N2—C6	−48.71 (19)	O4—Rb—O5—C24	−149.83 (14)
O5—Rb—N2—C5	63.11 (13)	O6—Rb—O5—C23	−172.65 (16)
O5—Rb—N2—C6	−103.42 (19)	O6—Rb—O5—C24	47.08 (12)
O6—Rb—N2—C5	0.43 (14)	N1—Rb—O6—C29	50.78 (14)
O6—Rb—N2—C6	−166.10 (17)	N1—Rb—O6—C30	−85.60 (16)
Rb—N1—C1—C2	173.77 (16)	N2—Rb—O6—C29	50.33 (15)
C5—N1—C1—C2	−0.8 (3)	N2—Rb—O6—C30	−86.05 (16)
N1—C1—C2—C3	0.4 (3)	O1—Rb—O6—C29	163.84 (15)
C1—C2—C3—C4	0.7 (3)	O1—Rb—O6—C30	27.46 (15)
C2—C3—C4—C5	−1.3 (3)	O2—Rb—O6—C29	−175.88 (13)
Rb—N2—C5—N1	0.33 (18)	O2—Rb—O6—C30	47.74 (16)
Rb—N2—C5—C4	176.26 (17)	O3—Rb—O6—C29	−121.55 (14)
C6—N2—C5—N1	169.51 (18)	O3—Rb—O6—C30	102.07 (16)
C6—N2—C5—C4	−14.6 (3)	O4—Rb—O6—C29	−62.94 (14)
Rb—N1—C5—N2	−0.32 (18)	O4—Rb—O6—C30	160.68 (15)
Rb—N1—C5—C4	−176.58 (15)	O5—Rb—O6—C29	−48.38 (13)
C1—N1—C5—N2	176.43 (18)	O5—Rb—O6—C30	175.24 (17)
C1—N1—C5—C4	0.2 (3)	Rb—O1—C12—C13	50.34 (19)
C3—C4—C5—N1	0.8 (3)	C31—O1—C12—C13	−174.83 (17)

C3—C4—C5—N2	−174.99 (19)	Rb—O2—C13—C12	47.05 (19)
Rb—N2—C6—C7	−59.5 (3)	C14—O2—C13—C12	−176.31 (17)
Rb—N2—C6—C11	114.2 (2)	O1—C12—C13—O2	−64.4 (2)
C5—N2—C6—C7	136.0 (2)	Rb—O2—C14—C15	137.31 (18)
C5—N2—C6—C11	−50.3 (3)	Rb—O2—C14—C19	−43.0 (2)
N2—C6—C7—C8	176.3 (2)	C13—O2—C14—C15	−1.3 (3)
C11—C6—C7—C8	2.1 (3)	C13—O2—C14—C19	178.36 (17)
C6—C7—C8—C9	−2.5 (3)	O2—C14—C15—C16	−178.7 (2)
C7—C8—C9—C10	0.9 (4)	C19—C14—C15—C16	1.6 (3)
C8—C9—C10—C11	0.9 (4)	C14—C15—C16—C17	−0.9 (4)
C9—C10—C11—C6	−1.2 (3)	C15—C16—C17—C18	−0.3 (4)
N2—C6—C11—C10	−174.1 (2)	C16—C17—C18—C19	0.8 (4)
C7—C6—C11—C10	−0.3 (3)	Rb—O3—C19—C14	45.6 (2)
N1—Rb—O1—C12	−149.39 (12)	Rb—O3—C19—C18	−135.05 (19)
N1—Rb—O1—C31	78.22 (15)	C20—O3—C19—C14	169.43 (19)
N2—Rb—O1—C12	−103.24 (13)	C20—O3—C19—C18	−11.2 (3)
N2—Rb—O1—C31	124.37 (15)	C17—C18—C19—O3	−179.4 (2)
O2—Rb—O1—C12	−18.03 (12)	C17—C18—C19—C14	−0.1 (3)
O2—Rb—O1—C31	−150.42 (16)	O2—C14—C19—O3	−1.4 (3)
O3—Rb—O1—C12	−2.63 (13)	O2—C14—C19—C18	179.19 (19)
O3—Rb—O1—C31	−135.02 (15)	C15—C14—C19—O3	178.31 (18)
O4—Rb—O1—C12	58.73 (14)	C15—C14—C19—C18	−1.1 (3)
O4—Rb—O1—C31	−73.66 (16)	Rb—O3—C20—C21	−68.17 (19)
O5—Rb—O1—C12	113.76 (12)	C19—O3—C20—C21	164.94 (19)
O5—Rb—O1—C31	−18.63 (16)	Rb—O4—C21—C20	−30.0 (2)
O6—Rb—O1—C12	139.33 (14)	C22—O4—C21—C20	−166.07 (18)
O6—Rb—O1—C31	6.94 (14)	O3—C20—C21—O4	68.4 (2)
N1—Rb—O2—C13	63.93 (14)	Rb—O4—C22—C23	55.22 (19)
N1—Rb—O2—C14	−74.70 (13)	C21—O4—C22—C23	−168.59 (17)
N2—Rb—O2—C13	107.82 (13)	Rb—O5—C23—C22	39.7 (2)
N2—Rb—O2—C14	−30.81 (12)	C24—O5—C23—C22	176.63 (18)
O1—Rb—O2—C13	−16.28 (11)	O4—C22—C23—O5	−61.3 (2)
O1—Rb—O2—C14	−154.91 (13)	Rb—O5—C24—C25	136.0 (2)
O3—Rb—O2—C13	−177.37 (14)	Rb—O5—C24—C29	−45.2 (2)
O3—Rb—O2—C14	44.00 (11)	C23—O5—C24—C25	−5.2 (3)
O4—Rb—O2—C13	−150.86 (12)	C23—O5—C24—C29	173.54 (18)
O4—Rb—O2—C14	70.51 (12)	O5—C24—C25—C26	179.6 (2)
O5—Rb—O2—C13	−93.99 (13)	C29—C24—C25—C26	0.9 (4)
O5—Rb—O2—C14	127.38 (12)	C24—C25—C26—C27	−0.2 (4)
O6—Rb—O2—C13	−36.68 (13)	C25—C26—C27—C28	0.0 (4)
O6—Rb—O2—C14	−175.32 (11)	C26—C27—C28—C29	−0.5 (4)
N1—Rb—O3—C19	71.09 (13)	Rb—O6—C29—C24	49.0 (2)
N1—Rb—O3—C20	−59.37 (14)	Rb—O6—C29—C28	−131.7 (2)
N2—Rb—O3—C19	66.71 (12)	C30—O6—C29—C24	−176.59 (19)
N2—Rb—O3—C20	−63.74 (13)	C30—O6—C29—C28	2.7 (3)
O1—Rb—O3—C19	−61.40 (13)	C27—C28—C29—O6	−178.0 (2)
O1—Rb—O3—C20	168.14 (13)	C27—C28—C29—C24	1.2 (4)
O2—Rb—O3—C19	−44.91 (12)	O5—C24—C29—O6	−0.9 (3)

O2—Rb—O3—C20	−175.37 (14)	O5—C24—C29—C28	179.8 (2)
O4—Rb—O3—C19	165.69 (14)	C25—C24—C29—O6	177.9 (2)
O4—Rb—O3—C20	35.24 (12)	C25—C24—C29—C28	−1.4 (3)
O5—Rb—O3—C19	−173.07 (12)	Rb—O6—C30—C31	−59.5 (2)
O5—Rb—O3—C20	56.48 (13)	C29—O6—C30—C31	167.46 (19)
O6—Rb—O3—C19	−119.57 (12)	Rb—O1—C31—C30	−38.8 (2)
O6—Rb—O3—C20	109.98 (13)	C12—O1—C31—C30	−173.43 (18)
N1—Rb—O4—C21	125.58 (15)	O6—C30—C31—O1	65.1 (2)
N1—Rb—O4—C22	−100.03 (14)		