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# *fac*-Trichloridotrispyrazolerhodium(III)

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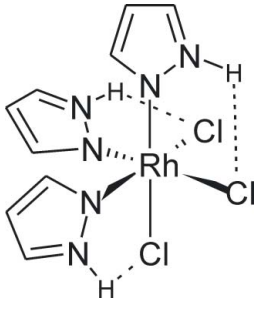
Key indicators: single-crystal X-ray study; $T$ = 108 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.032; $wR$ factor = 0.081; data-to-parameter ratio = 18.8.

The title compound, *fac*-[$RhCl_3(pzH)_3$] (pzH = pyrazole) or *fac*-[$RhCl_3(C_3H_4N_2)_3$], was prepared by treating $RhCl_3{\cdot}3H_2O$ with three equivalents of pyrazole. It contains a central $Rh^{III}$ atom coordinated in a facial manner by three pyrazole N atoms and three chloride ligands. As a result of their participation in weak intramolecular N—H···Cl hydrogen bonds, the pyrazole rings adopt a propeller-like arrangement. The mean planes of the pyrazole ligands exhibit dihedral angles of 26.83 (17), 2.03 (10) and 18.83 (14)° with the planes containing their coordinating N atoms, the $Rh^{III}$ atom and the acceptor Cl atoms of their individual N—H···Cl interactions.

## Related literature

For related literature, see: Albinati *et al.* (1999); Cushing *et al.* (2006); Nicto *et al.* (2005).




## Experimental

### *Crystal data*

$[RhCl_3(C_3H_4N_2)_3]$
$M_r$ = 413.51
Monoclinic, $P2_1/n$
$a$ = 8.2041 (3) Å
$b$ = 11.9243 (4) Å
$c$ = 14.5839 (5) Å
$\beta$ = 100.624 (3)°
$V$ = 1402.26 (8) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 1.78 mm$^{-1}$
$T$ = 108 (2) K
0.52 × 0.18 × 0.13 mm

### *Data collection*

Oxford Diffraction Sapphire2 CCD diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
$T_{min}$ = 0.692, $T_{max}$ = 0.802
25826 measured reflections
3236 independent reflections
2701 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.041

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.081
$S$ = 1.05
3236 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.88 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.78 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N32—H32···Cl1 | 0.86 | 2.54 | 3.053 (3) | 119 |
| N12—H12···Cl2 | 0.86 | 2.70 | 3.127 (3) | 112 |
| N22—H22···Cl3 | 0.86 | 2.46 | 3.017 (3) | 123 |
| C15—H15···Cl2$^{i}$ | 0.93 | 2.64 | 3.330 (3) | 131 |
| C33—H33···Cl2$^{ii}$ | 0.93 | 2.69 | 3.471 (3) | 142 |
| C24—H24···Cl3$^{iii}$ | 0.93 | 2.76 | 3.688 (3) | 178 |
| N22—H22···Cl1$^{iv}$ | 0.86 | 2.88 | 3.472 (3) | 128 |

Symmetry codes: (i) $-x-\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x-\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$; (iii) $x-\frac{1}{2}, -y+\frac{5}{2}, z-\frac{1}{2}$; (iv) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1995); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BV2069).

## References

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# supplementary materials

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# *fac*-Trichloridotrispyrazolerhodium(III)

**R. Bieda, M. C. Winter and W. S. Sheldrick**

## Comment

The complexes *mer*-[$MCl_3(Me_2pzH)_3$] ·$CH_3OH$ (*M* = Rh, Ir; $Me_2pzH$ = 3,5-dimethylpyrazole) have been prepared by treating $MCl_3$ ·$xH_2O$ with three equivalents of 3,5-dimethylpyrazole in methanol at 85°C (Cushing *et al.*, 2006). Only the *mer*-isomers were obtained on subsequent recrystallization from methanol solution. *mer*-[$RhCl_3(Me_2pzH)_3$] has also been characterized as a decomposition product of the reaction of [$RhCl_3(CH_3CN)_3$] with Tl[$HB(Me_2pz)]_3$ (Albinati *et al.*, 1999). A facial tris(dimethylpyrazole) $Re^I$ complex *fac*-[$Re(CO)_3(Me_2pzH)_3$][$B(Ar)_4$] (Ar = 3,5-bis(trifluoromethylphenyl)) is also known (Nicto *et al.*, 2005).

We now report the crystal structure of *fac*-[$RhCl_3(pzH)_3$] (I), which was obtained by treating $RhCl_3$ ·$3H_2O$ in a 2-propanol/water mixture with three equivalents of pyrazole. (I) was recrystallized as the *fac*-isomer from methanol solution and exhibits the expected $^1$H NMR resonances at 6.57 (*t*), 6.88 (*d*) and 7.89 (*d*) for the C—H protons and 12.48 (*d*) for the N—H protons of the pure isomer in $CDCl_3$ solution. A 2:1 mixture of the *fac* and *mer* isomers is observed after 5 h in methanol solution. As depicted in Fig. 1, the pyrazole ligands adopt a propellor-like arrangement in (I) and participate in intramolecular N—H···Cl hydrogen bonds (Table 1) to the nearest chloride ligand. Their best planes exhibit respective dihedral angles of 26.83 (17), 2.03 (10) and 18.83 (14)° to the planes containing their coordinating nitrogen atoms, Rh1 and the acceptor Cl atoms of their N—H···Cl interactions. The molecules are linked into a network by intermolecular N—H···Cl and C—H···Cl hydrogen bonds (Fig. 2).

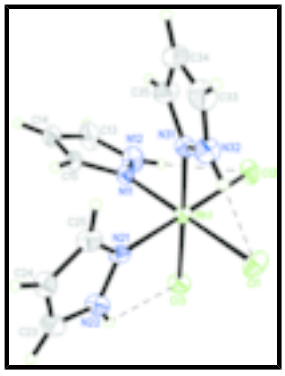
## Experimental

*fac*-[$RhCl_3(pzH)_3$] (I) was prepared by treating 50.5 mg $RhCl_3$ ·$3H_2O$ (0.19 mmol) in 0.9 ml 2-propanol/water (5:1) with 3 equivalents of pyrazole (40.2 mg, 0.59 mmol). After stirring for 1.5 h, the resulting yellow precipitate was removed by centrifugation and washed with 2 ml of 2-propanol to afford *fac*-[$RhCl_3(pzH)_3$] in 17% yield (13.7 mg). Elemental anlysis (Vario EL) for $C_9H_{12}Cl_3N_6Rh$ ·$H_2O$ (*M* = 431.5): C 25.4 (calc. 25.7), H 3.2 (calc. 3.4), N 19.4 (calc. 19.3). FAB MS on a VG Autospec: m/*z* 435(3%) $[M+Na]^+$, 377(15%) $[M—Cl]^+$. $^1$H NMR ($CDCl_3$) on a Bruker DRX 400: δ 6.57 (t, 3H, pzH-b), 6.88 (d, 3H, pzH-c), 7.89 (d, 3H, pyH-a), 12.48 (d, 3H, NH). Suitable crystals for X-ray analysis were obtained by slow evaporation of a methanol solution of (I).

## Refinement

H atoms were constrained to idealized positions and refined using a riding model, with C—H distances of 0.93 Å and N—H distances of 0.86 Å; $U_{iso}$(H) = 1.2 $U_{iso}$(C) for the former and $U_{iso}$(H) = 1.2 $U_{iso}$(N) for the latter protons.

### Figures



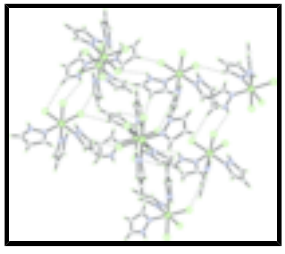


Fig. 1 Molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level. Fig. 2 Hydrogen bonds between the anions and cations of (I). The colour code is the same as in Fig. 1.

### *fac*-Trichloridotrispyrazolerhodium(III)

*Crystal data*

| | |
|---|---|
| $[RhCl_3(C_3H_4N_2)_3]$ | $F_{000}$ = 816 |
| $M_r$ = 413.51 | $D_x$ = 1.959 Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| Hall symbol: -P 2yn | Cell parameters from 14706 reflections |
| $a$ = 8.2041 (3) Å | θ = 2.5–27.5º |
| $b$ = 11.9243 (4) Å | μ = 1.78 mm$^{-1}$ |
| $c$ = 14.5839 (5) Å | $T$ = 108 (2) K |
| β = 100.624 (3)º | Prismatic, orange |
| $V$ = 1402.26 (8) Å$^3$ | 0.52 × 0.18 × 0.13 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Oxford Diffraction Sapphire2 CCD diffractometer | 3236 independent reflections |
| Radiation source: fine-focus sealed tube | 2701 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.041 |
| $T$ = 108(2) K | $\theta_{max}$ = 27.6º |
| 1109 images at 1.0 deg in ω and 20 s scans | $\theta_{min}$ = 2.8º |
| Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2006) | $h$ = −10→10 |
| $T_{min}$ = 0.692, $T_{max}$ = 0.802 | $k$ = −15→14 |

25826 measured reflections

$l = -19 \rightarrow 18$

*Refinement*

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.081$

$S = 1.05$

3236 reflections

172 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0483P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.88$ e Å$^{-3}$

$\Delta\rho_{min} = -0.78$ e Å$^{-3}$

Extinction correction: none

*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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Rh1 | −0.00400 (3) | 0.990704 (18) | 0.245999 (14) | 0.01730 (9) |
| Cl1 | 0.19460 (10) | 0.92389 (8) | 0.16155 (5) | 0.03188 (19) |
| Cl2 | 0.01555 (10) | 0.82640 (6) | 0.33384 (5) | 0.02876 (18) |
| Cl3 | 0.19940 (9) | 1.07268 (7) | 0.35894 (5) | 0.02689 (17) |
| N11 | −0.1773 (3) | 1.0492 (2) | 0.31679 (17) | 0.0195 (5) |
| N12 | −0.1894 (4) | 1.0192 (2) | 0.40354 (19) | 0.0319 (6) |
| H12 | −0.1245 | 0.9729 | 0.4377 | 0.038* |
| C13 | −0.3195 (5) | 1.0729 (3) | 0.4296 (2) | 0.0358 (8) |
| H13 | −0.3541 | 1.0660 | 0.4866 | 0.043* |
| C14 | −0.3900 (4) | 1.1389 (3) | 0.3568 (2) | 0.0290 (7) |
| H14 | −0.4817 | 1.1855 | 0.3540 | 0.035* |
| C15 | −0.2986 (4) | 1.1227 (2) | 0.2887 (2) | 0.0201 (6) |
| H15 | −0.3182 | 1.1580 | 0.2309 | 0.024* |
| N21 | −0.0229 (3) | 1.1355 (2) | 0.16938 (17) | 0.0211 (5) |
| N22 | 0.0733 (3) | 1.2255 (2) | 0.19646 (18) | 0.0258 (6) |
| H22 | 0.1480 | 1.2284 | 0.2463 | 0.031* |
| C23 | 0.0374 (4) | 1.3100 (3) | 0.1356 (2) | 0.0278 (7) |

| | | | | |
|---|---|---|---|---|
| H23 | 0.0873 | 1.3803 | 0.1398 | 0.033* |
| C24 | −0.0870 (4) | 1.2733 (3) | 0.0658 (2) | 0.0271 (7) |
| H24 | −0.1381 | 1.3130 | 0.0135 | 0.033* |
| C25 | −0.1203 (4) | 1.1638 (3) | 0.0903 (2) | 0.0267 (7) |
| H25 | −0.1997 | 1.1173 | 0.0558 | 0.032* |
| N31 | −0.1879 (3) | 0.9203 (2) | 0.14972 (17) | 0.0208 (5) |
| N32 | −0.1648 (4) | 0.8932 (2) | 0.06342 (18) | 0.0292 (6) |
| H32 | −0.0731 | 0.9016 | 0.0436 | 0.035* |
| C33 | −0.3044 (5) | 0.8512 (3) | 0.0127 (2) | 0.0390 (9) |
| H33 | −0.3184 | 0.8265 | −0.0488 | 0.047* |
| C34 | −0.4233 (4) | 0.8513 (3) | 0.0680 (3) | 0.0379 (8) |
| H34 | −0.5325 | 0.8268 | 0.0520 | 0.045* |
| C35 | −0.3450 (4) | 0.8960 (3) | 0.1528 (2) | 0.0297 (7) |
| H35 | −0.3952 | 0.9073 | 0.2043 | 0.036* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Rh1 | 0.01787 (13) | 0.02047 (13) | 0.01265 (12) | 0.00115 (8) | 0.00043 (8) | 0.00065 (8) |
| Cl1 | 0.0259 (4) | 0.0454 (5) | 0.0252 (4) | 0.0060 (3) | 0.0069 (3) | −0.0037 (3) |
| Cl2 | 0.0346 (4) | 0.0227 (4) | 0.0259 (4) | 0.0059 (3) | −0.0025 (3) | 0.0035 (3) |
| Cl3 | 0.0261 (4) | 0.0300 (4) | 0.0206 (3) | −0.0029 (3) | −0.0061 (3) | 0.0014 (3) |
| N11 | 0.0220 (12) | 0.0179 (12) | 0.0172 (11) | −0.0001 (9) | 0.0001 (9) | 0.0013 (9) |
| N12 | 0.0423 (17) | 0.0324 (15) | 0.0203 (13) | 0.0064 (13) | 0.0040 (12) | 0.0056 (11) |
| C13 | 0.053 (2) | 0.0321 (18) | 0.0272 (17) | 0.0060 (16) | 0.0211 (16) | 0.0030 (14) |
| C14 | 0.0290 (17) | 0.0246 (16) | 0.0361 (18) | 0.0027 (13) | 0.0126 (14) | −0.0006 (13) |
| C15 | 0.0221 (14) | 0.0204 (14) | 0.0174 (13) | 0.0029 (11) | 0.0021 (11) | 0.0038 (11) |
| N21 | 0.0177 (12) | 0.0254 (13) | 0.0198 (12) | −0.0026 (10) | 0.0025 (9) | 0.0007 (10) |
| N22 | 0.0267 (13) | 0.0303 (14) | 0.0189 (12) | −0.0040 (11) | 0.0003 (10) | 0.0018 (10) |
| C23 | 0.0324 (17) | 0.0286 (17) | 0.0237 (15) | −0.0048 (13) | 0.0084 (13) | 0.0039 (12) |
| C24 | 0.0305 (16) | 0.0307 (17) | 0.0199 (14) | 0.0027 (13) | 0.0040 (12) | 0.0066 (12) |
| C25 | 0.0267 (16) | 0.0323 (17) | 0.0197 (14) | −0.0022 (13) | 0.0010 (12) | 0.0024 (12) |
| N31 | 0.0238 (12) | 0.0207 (12) | 0.0167 (11) | −0.0001 (10) | 0.0006 (9) | 0.0000 (9) |
| N32 | 0.0337 (15) | 0.0347 (15) | 0.0187 (12) | −0.0005 (12) | 0.0036 (11) | −0.0052 (11) |
| C33 | 0.044 (2) | 0.040 (2) | 0.0280 (18) | 0.0013 (16) | −0.0076 (15) | −0.0123 (15) |
| C34 | 0.0285 (18) | 0.038 (2) | 0.042 (2) | −0.0012 (15) | −0.0078 (15) | −0.0092 (16) |
| C35 | 0.0249 (16) | 0.0360 (18) | 0.0275 (16) | −0.0022 (13) | 0.0026 (13) | −0.0042 (14) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Rh1—N11 | 2.029 (3) | N22—C23 | 1.339 (4) |
| Rh1—N31 | 2.043 (2) | N22—H22 | 0.8600 |
| Rh1—N21 | 2.047 (3) | C23—C24 | 1.373 (5) |
| Rh1—Cl2 | 2.3302 (8) | C23—H23 | 0.9300 |
| Rh1—Cl3 | 2.3323 (7) | C24—C25 | 1.394 (5) |
| Rh1—Cl1 | 2.3550 (8) | C24—H24 | 0.9300 |
| N11—C15 | 1.333 (4) | C25—H25 | 0.9300 |
| N11—N12 | 1.336 (4) | N31—C35 | 1.330 (4) |
| N12—C13 | 1.358 (5) | N31—N32 | 1.346 (3) |

| | | | |
|---|---|---|---|
| N12—H12 | 0.8600 | N32—C33 | 1.341 (4) |
| C13—C14 | 1.363 (5) | N32—H32 | 0.8600 |
| C13—H13 | 0.9300 | C33—C34 | 1.375 (6) |
| C14—C15 | 1.363 (4) | C33—H33 | 0.9300 |
| C14—H14 | 0.9300 | C34—C35 | 1.392 (5) |
| C15—H15 | 0.9300 | C34—H34 | 0.9300 |
| N21—C25 | 1.320 (4) | C35—H35 | 0.9300 |
| N21—N22 | 1.348 (4) | | |
| N11—Rh1—N31 | 89.60 (10) | C25—N21—N22 | 106.3 (3) |
| N11—Rh1—N21 | 89.87 (10) | C25—N21—Rh1 | 131.7 (2) |
| N31—Rh1—N21 | 89.96 (10) | N22—N21—Rh1 | 122.04 (18) |
| N11—Rh1—Cl2 | 89.61 (7) | C23—N22—N21 | 111.0 (3) |
| N31—Rh1—Cl2 | 89.98 (7) | C23—N22—H22 | 124.5 |
| N21—Rh1—Cl2 | 179.48 (7) | N21—N22—H22 | 124.5 |
| N11—Rh1—Cl3 | 88.49 (7) | N22—C23—C24 | 107.1 (3) |
| N31—Rh1—Cl3 | 178.08 (7) | N22—C23—H23 | 126.4 |
| N21—Rh1—Cl3 | 90.25 (7) | C24—C23—H23 | 126.4 |
| Cl2—Rh1—Cl3 | 89.79 (3) | C23—C24—C25 | 105.2 (3) |
| N11—Rh1—Cl1 | 179.07 (7) | C23—C24—H24 | 127.4 |
| N31—Rh1—Cl1 | 89.77 (7) | C25—C24—H24 | 127.4 |
| N21—Rh1—Cl1 | 89.43 (7) | N21—C25—C24 | 110.3 (3) |
| Cl2—Rh1—Cl1 | 91.08 (3) | N21—C25—H25 | 124.8 |
| Cl3—Rh1—Cl1 | 92.14 (3) | C24—C25—H25 | 124.8 |
| C15—N11—N12 | 106.6 (3) | C35—N31—N32 | 106.5 (3) |
| C15—N11—Rh1 | 128.6 (2) | C35—N31—Rh1 | 131.6 (2) |
| N12—N11—Rh1 | 124.8 (2) | N32—N31—Rh1 | 121.9 (2) |
| N11—N12—C13 | 109.8 (3) | C33—N32—N31 | 110.6 (3) |
| N11—N12—H12 | 125.1 | C33—N32—H32 | 124.7 |
| C13—N12—H12 | 125.1 | N31—N32—H32 | 124.7 |
| N12—C13—C14 | 107.2 (3) | N32—C33—C34 | 107.6 (3) |
| N12—C13—H13 | 126.4 | N32—C33—H33 | 126.2 |
| C14—C13—H13 | 126.4 | C34—C33—H33 | 126.2 |
| C13—C14—C15 | 105.9 (3) | C33—C34—C35 | 105.1 (3) |
| C13—C14—H14 | 127.0 | C33—C34—H34 | 127.4 |
| C15—C14—H14 | 127.0 | C35—C34—H34 | 127.4 |
| N11—C15—C14 | 110.5 (3) | N31—C35—C34 | 110.2 (3) |
| N11—C15—H15 | 124.8 | N31—C35—H35 | 124.9 |
| C14—C15—H15 | 124.8 | C34—C35—H35 | 124.9 |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N32—H32···Cl1 | 0.86 | 2.54 | 3.053 (3) | 119 |
| N12—H12···Cl2 | 0.86 | 2.70 | 3.127 (3) | 112 |
| N22—H22···Cl3 | 0.86 | 2.46 | 3.017 (3) | 123 |
| C15—H15···Cl2$^{i}$ | 0.93 | 2.64 | 3.330 (3) | 131 |
| C33—H33···Cl2$^{ii}$ | 0.93 | 2.69 | 3.471 (3) | 142 |
| C24—H24···Cl3$^{iii}$ | 0.93 | 2.76 | 3.688 (3) | 178 |

| | | | | |
|---|---|---|---|---|
| N22—H22···Cl1$^{iv}$ | 0.86 | 2.88 | 3.472 (3) | 128 |

Symmetry codes: (i) −*x*−1/2, *y*+1/2, −*z*+1/2; (ii) *x*−1/2, −*y*+3/2, *z*−1/2; (iii) *x*−1/2, −*y*+5/2, *z*−1/2; (iv) −*x*+1/2, *y*+1/2, −*z*+1/2.

**Fig. 1**

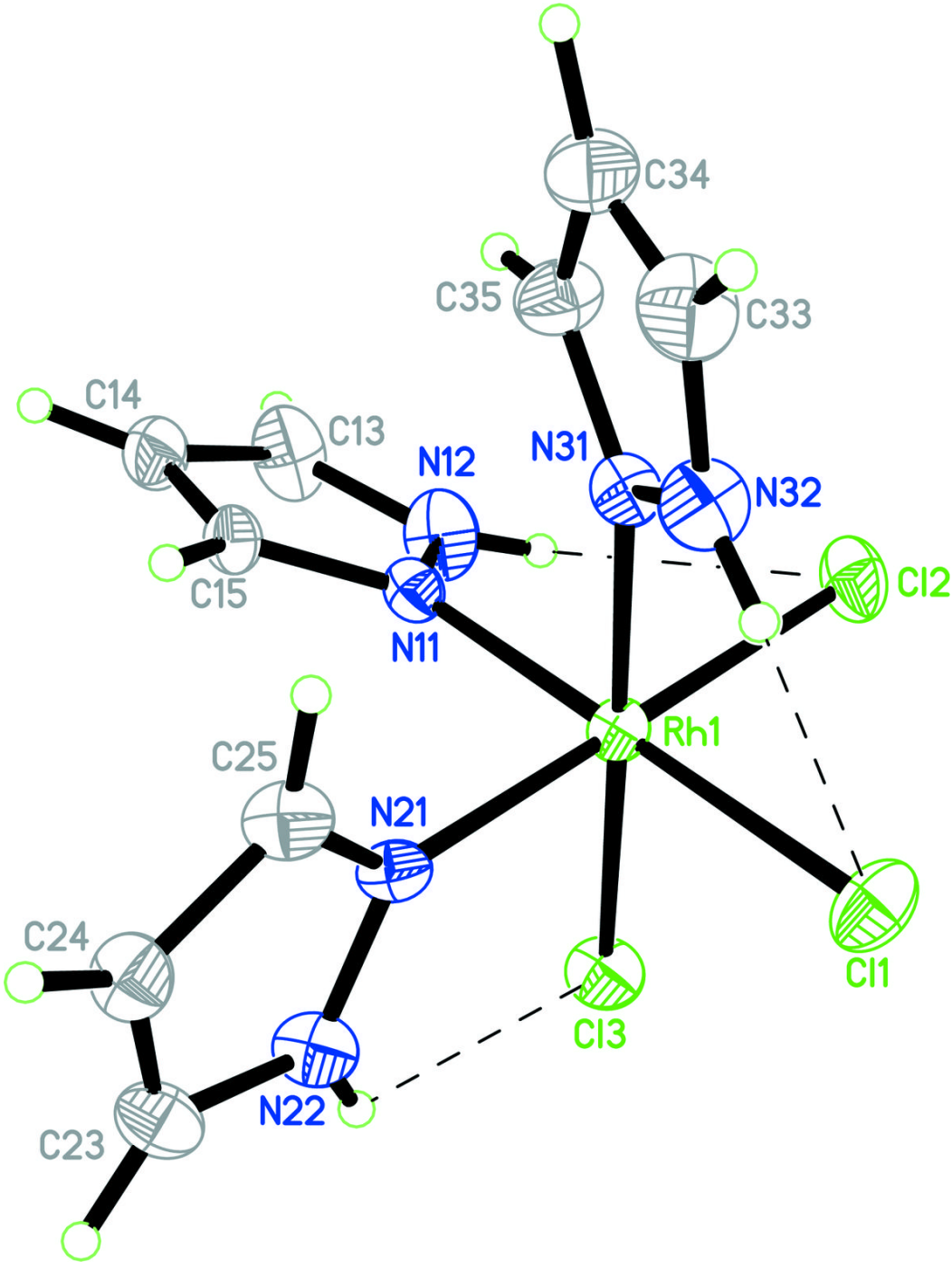


**Fig. 2**