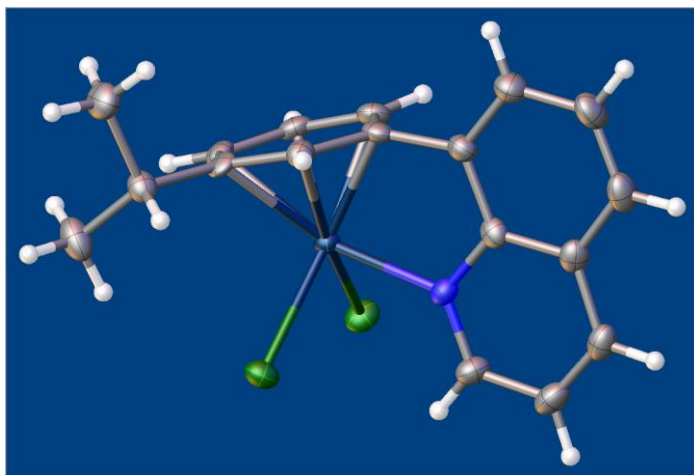


Sample ID: 04151

 **$R_1 = 2.44\%$** 

## Crystal Data and Experimental



**Experimental.** Single clear pale yellow needle-shaped crystals of **04151** were obtained. A suitable crystal  $0.09 \times 0.03 \times 0.01$  mm<sup>3</sup> was selected and placed on a MiTeGen micromount on an XtaLAB Synergy R, HyPix-Arc 100 diffractometer. The crystal was kept at a steady  $T = 150.00(10)$  K during data collection. The structure was solved with the ShelXT 2018/2 (Sheldrick, 2018) structure solution program using the Intrinsic Phasing solution method and by using Olex2 (Dolomanov et al., 2009) as the graphical interface. The model was refined with version 2018/3 of ShelXL 2018/3 (Sheldrick, 2015) using Least Squares minimisation.

**Crystal Data.** C<sub>18</sub>H<sub>17</sub>NCl<sub>2</sub>O<sub>s</sub>,  $M_r = 508.42$ , monoclinic,  $P2_1/c$  (No. 14),  $a = 10.3372(2)$  Å,  $b = 16.3941(3)$  Å,  $c = 9.7316(2)$  Å,  $\beta = 102.969(2)^\circ$ ,  $\alpha = \gamma = 90^\circ$ ,  $V = 1607.14(6)$  Å<sup>3</sup>,  $T = 150.00(10)$  K,  $Z = 4$ ,  $Z' = 1$ ,  $\mu(\text{Cu K}\alpha) = 17.990$ , 27764 reflections measured, 2934 unique ( $R_{\text{int}} = 0.0565$ ) which were used in all calculations. The final  $wR_2$  was 0.0661 (all data) and  $R_1$  was 0.0244 ( $I > 2(I)$ ).

Compound	04151
Formula	C <sub>18</sub> H <sub>17</sub> NCl <sub>2</sub> O <sub>s</sub>
$D_{\text{calc}} / \text{g cm}^{-3}$	2.101
$\mu / \text{mm}^{-1}$	17.990
Formula Weight	508.42
Colour	clear pale yellow
Shape	needle
Size/mm <sup>3</sup>	$0.09 \times 0.03 \times 0.01$
$T/\text{K}$	150.00(10)
Crystal System	monoclinic
Space Group	$P2_1/c$
$a/\text{\AA}$	10.3372(2)
$b/\text{\AA}$	16.3941(3)
$c/\text{\AA}$	9.7316(2)
$\alpha/^\circ$	90
$\beta/^\circ$	102.969(2)
$\gamma/^\circ$	90
$V/\text{\AA}^3$	1607.14(6)
$Z$	4
$Z'$	1
Wavelength/Å	1.54184
Radiation type	Cu K $\alpha$
$\theta_{\text{min}}/^\circ$	4.389
$\theta_{\text{max}}/^\circ$	68.429
Measured Refl.	27764
Independent Refl.	2934
Reflections with $I > 2(I)$	2715
$R_{\text{int}}$	0.0565
Parameters	201
Restraints	0
Largest Peak	0.980
Deepest Hole	-1.305
GooF	1.067
$wR_2$ (all data)	0.0661
$wR_2$	0.0651
$R_1$ (all data)	0.0267
$R_1$	0.0244

Structure Quality Indicators

Reflections:	d min (CuKα) 2θ=136.9°	0.83	I/σ(I)	38.8	Rint m=9.69	5.65%	Full 135.4° 99% to 136.9°	99.8
	Shift	-0.002	Max Peak	1.0	Min Peak	-1.3	Goof	1.067

**Experimental Extended.** A clear pale yellow needle-shaped crystal with dimensions 0.09×0.03×0.01 mm<sup>3</sup> was placed on a MiTeGen micromount. Data were collected using an XtaLAB Synergy R, HyPix-Arc 100 diffractometer operating at *T* = 150.00(10) K.

Data were measured using  $\omega$  scans of 0.5° per frame for 0.2/0.1 s using Cu K $\alpha$  radiation. The diffraction pattern was indexed and the total number of runs and images was based on the strategy calculation from the program CrysAlisPro (Rigaku) The maximum resolution that was achieved was  $\Theta$  = 68.429° (0.83 Å).

The diffraction pattern was indexed The diffraction pattern was indexed and the total number of runs and images was based on the strategy calculation from the program CrysAlisPro (Rigaku) and the unit cell was refined using CrysAlisPro (Rigaku, V1.171.43.127a, 2024) on 17453 reflections, 63% of the observed reflections.

Data reduction, scaling and absorption corrections were performed using CrysAlisPro (Rigaku, V1.171.43.127a, 2024). The final completeness is 99.80 % out to 68.429° in  $\Theta$ . A gaussian absorption correction was performed using CrysAlisPro 1.171.43.127a (Rigaku Oxford Diffraction, 2024) Numerical absorption correction based on gaussian integration over a multifaceted crystal model Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm. The absorption coefficient  $\mu$  of this material is 17.990 mm<sup>-1</sup> at this wavelength ( $\lambda$  = 1.542Å) and the minimum and maximum transmissions are 0.731 and 0.978.

The structure was solved and the space group *P*2<sub>1</sub>/*c* (# 14) determined by the ShelXT 2018/2 (Sheldrick, 2018) structure solution program using Intrinsic Phasing and refined by Least Squares using version 2018/3 of ShelXL 2018/3 (Sheldrick, 2015). All non-hydrogen atoms were refined anisotropically. Hydrogen atom positions were calculated geometrically and refined using the riding model. Hydrogen atom positions were calculated geometrically and refined using the riding model.

*\_exptl\_absorpt\_process\_details*: CrysAlisPro 1.171.43.127a (Rigaku Oxford Diffraction, 2024) Numerical absorption correction based on gaussian integration over a multifaceted crystal model Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

**Table 1:** Fractional Atomic Coordinates (×10<sup>4</sup>) and Equivalent Isotropic Displacement Parameters (Å<sup>2</sup>×10<sup>3</sup>) for **04151**. *U*<sub>eq</sub> is defined as 1/3 of the trace of the orthogonalised *U*<sub>ij</sub>.

Atom	x	y	z	<i>U</i> <sub>eq</sub>
Os1	7272.2(2)	6302.0(2)	6361.2(2)	17.28(9)
Cl1	6981.3(10)	5620.9(6)	4122.4(10)	26.2(2)
Cl2	9355.6(9)	6773.3(6)	5940.1(11)	26.2(2)
N1	8340(3)	5252(2)	7267(3)	20.3(7)
C1	5442(4)	6042(3)	7040(4)	21.6(8)
C2	6551(4)	6169(3)	8203(4)	22.0(8)
C3	7267(4)	6925(3)	8323(4)	23.7(8)
C4	6958(4)	7497(3)	7201(4)	24.7(8)
C5	5899(4)	7355(2)	6011(4)	22.3(8)
C6	5114(3)	6636(3)	5936(4)	20.8(8)
C7	7139(4)	5457(3)	9075(4)	22.6(8)
C8	6861(4)	5230(3)	10350(4)	29.5(9)

Atom	x	y	z	$U_{eq}$
C9	7510(5)	4548(3)	11085(5)	33.9(10)
C10	8410(4)	4105(3)	10564(4)	31.7(9)
C11	8726(4)	4321(2)	9269(4)	25.2(8)
C12	9644(4)	3889(3)	8656(5)	28.2(9)
C13	9893(4)	4151(3)	7418(5)	30.6(9)
C14	9221(4)	4843(3)	6744(4)	26.1(9)
C15	8080(4)	5004(3)	8535(4)	21.9(8)
C16	3933(4)	6476(3)	4740(4)	25.0(8)
C17	2709(4)	6805(3)	5217(5)	35.5(10)
C18	4041(5)	6857(3)	3343(5)	36.7(11)

**Table 2:** Anisotropic Displacement Parameters ( $\times 10^4$ ) **04151**. The anisotropic displacement factor exponent takes the form:  $-2\pi^2[h^2a^{*2} \times U_{11} + \dots + 2hka^* \times b^* \times U_{12}]$

Atom	$U_{11}$	$U_{22}$	$U_{33}$	$U_{23}$	$U_{13}$	$U_{12}$
Os1	16.31(12)	17.91(13)	18.71(12)	-0.59(6)	6.26(8)	-0.94(5)
Cl1	31.5(5)	27.2(5)	21.6(4)	-2.9(4)	9.5(4)	-3.1(4)
Cl2	19.4(4)	25.6(5)	36.1(5)	1.5(4)	11.3(4)	-3.7(3)
N1	17.0(15)	20.0(16)	23.6(16)	-0.7(13)	4.4(13)	1.1(12)
C1	20.2(19)	24(2)	22.5(19)	3.8(17)	8.4(15)	1.4(16)
C2	22(2)	29(2)	17.6(18)	-5.2(16)	9.3(16)	0.2(16)
C3	27(2)	24(2)	22.7(18)	-7.3(16)	10.1(16)	-0.6(16)
C4	24(2)	21(2)	32(2)	-5.2(16)	12.7(17)	3.3(15)
C5	17.9(18)	24(2)	27(2)	2.7(16)	9.2(15)	7.5(15)
C6	10.4(17)	27(2)	27(2)	0.5(17)	8.0(15)	4.6(15)
C7	22.5(19)	27(2)	18.8(18)	-1.7(16)	5.2(15)	-0.6(15)
C8	31(2)	33(2)	26(2)	0.8(18)	10.7(18)	1.0(18)
C9	45(3)	33(3)	22.5(19)	5.0(18)	6.7(19)	-1(2)
C10	40(2)	24(2)	27(2)	4.4(17)	-1.6(18)	-2.8(18)
C11	28(2)	18(2)	28(2)	-1.8(16)	0.9(16)	-1.6(16)
C12	25(2)	21(2)	34(2)	-0.8(18)	-1.8(18)	1.2(17)
C13	25(2)	25(2)	41(2)	-5.5(19)	6.3(18)	3.8(17)
C14	26(2)	24(2)	31(2)	-1.4(17)	11.3(17)	1.9(16)
C15	17.5(17)	26(2)	20.5(18)	0.0(16)	1.6(15)	-4.3(16)
C16	22(2)	30(2)	23(2)	3.2(17)	5.6(16)	-2.8(17)
C17	23(2)	45(3)	36(2)	-2(2)	2.3(19)	0.4(19)
C18	32(2)	49(3)	27(2)	8(2)	0.6(18)	-10(2)

**Table 3:** Bond Lengths in Å for **04151**.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Os1	Cl1	2.4062(9)	C4	C5	1.423(6)
Os1	Cl2	2.4082(9)	C5	C6	1.423(6)
Os1	N1	2.126(3)	C6	C16	1.509(6)
Os1	C1	2.182(4)	C7	C8	1.386(6)
Os1	C2	2.101(4)	C7	C15	1.416(6)
Os1	C3	2.166(4)	C8	C9	1.413(6)
Os1	C4	2.175(4)	C9	C10	1.364(7)
Os1	C5	2.212(4)	C10	C11	1.416(6)
Os1	C6	2.244(3)	C11	C12	1.419(6)
N1	C14	1.321(5)	C11	C15	1.413(6)
N1	C15	1.381(5)	C12	C13	1.357(7)
C1	C2	1.434(6)	C13	C14	1.413(6)
C1	C6	1.434(6)	C16	C17	1.539(6)
C2	C3	1.435(6)	C16	C18	1.522(6)
C2	C7	1.491(6)			
C3	C4	1.420(6)			

**Table 4:** Bond Angles in ° for **04151**.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
Cl1	Os1	Cl2	85.97(3)	C6	C1	C2	120.3(4)
N1	Os1	Cl1	87.00(9)	C1	C2	Os1	73.5(2)
N1	Os1	Cl2	85.95(9)	C1	C2	C3	119.8(4)
N1	Os1	C1	96.97(14)	C1	C2	C7	119.1(4)
N1	Os1	C3	96.90(14)	C3	C2	Os1	72.8(2)
N1	Os1	C4	132.90(14)	C3	C2	C7	119.5(4)
N1	Os1	C5	161.98(13)	C7	C2	Os1	112.9(3)
N1	Os1	C6	132.56(13)	C2	C3	Os1	67.9(2)
C1	Os1	Cl1	104.06(11)	C4	C3	Os1	71.3(2)
C1	Os1	Cl2	169.66(11)	C4	C3	C2	119.0(4)
C1	Os1	C5	68.20(15)	C3	C4	Os1	70.6(2)
C1	Os1	C6	37.77(15)	C3	C4	C5	121.1(4)
C2	Os1	Cl1	136.73(11)	C5	C4	Os1	72.5(2)
C2	Os1	Cl2	132.88(11)	C4	C5	Os1	69.7(2)
C2	Os1	N1	79.24(14)	C6	C5	Os1	72.6(2)
C2	Os1	C1	39.06(15)	C6	C5	C4	120.2(4)
C2	Os1	C3	39.26(16)	C1	C6	Os1	68.8(2)
C2	Os1	C4	70.18(16)	C1	C6	C16	118.3(4)
C2	Os1	C5	82.75(15)	C5	C6	Os1	70.2(2)
C2	Os1	C6	69.76(15)	C5	C6	C1	119.2(4)
C3	Os1	Cl1	172.87(11)	C5	C6	C16	122.5(4)
C3	Os1	Cl2	100.23(11)	C16	C6	Os1	134.3(3)
C3	Os1	C1	69.61(15)	C8	C7	C2	125.5(4)
C3	Os1	C4	38.17(16)	C8	C7	C15	119.4(4)
C3	Os1	C5	68.87(15)	C15	C7	C2	115.2(3)
C3	Os1	C6	81.29(15)	C7	C8	C9	119.7(4)
C4	Os1	Cl1	139.40(11)	C10	C9	C8	121.3(4)
C4	Os1	Cl2	89.42(11)	C9	C10	C11	120.7(4)
C4	Os1	C1	81.32(15)	C10	C11	C12	124.0(4)
C4	Os1	C5	37.85(15)	C15	C11	C10	118.1(4)
C4	Os1	C6	67.85(15)	C15	C11	C12	117.9(4)
C5	Os1	Cl1	106.07(11)	C13	C12	C11	119.5(4)
C5	Os1	Cl2	106.92(11)	C12	C13	C14	119.6(4)
C5	Os1	C6	37.23(15)	N1	C14	C13	122.6(4)
C6	Os1	Cl1	91.67(10)	N1	C15	C7	117.7(4)
C6	Os1	Cl2	141.30(11)	N1	C15	C11	121.4(4)
C14	N1	Os1	126.1(3)	C11	C15	C7	120.9(4)
C14	N1	C15	118.9(3)	C6	C16	C17	106.5(3)
C15	N1	Os1	114.9(3)	C6	C16	C18	114.1(3)
C2	C1	Os1	67.4(2)	C18	C16	C17	110.9(4)
C6	C1	Os1	73.5(2)				

**Table 5:** Torsion Angles in ° for **04151**.

Atom	Atom	Atom	Atom	Angle/°
Os1	N1	C14	C13	179.7(3)
Os1	N1	C15	C7	0.9(4)
Os1	N1	C15	C11	-179.3(3)
Os1	C1	C2	C3	58.4(3)
Os1	C1	C2	C7	-107.5(3)
Os1	C1	C6	C5	-50.6(3)
Os1	C1	C6	C16	130.0(3)
Os1	C2	C3	C4	51.6(3)
Os1	C2	C7	C8	179.1(3)
Os1	C2	C7	C15	0.8(4)
Os1	C3	C4	C5	54.1(3)
Os1	C4	C5	C6	54.2(3)
Os1	C5	C6	C1	50.0(3)
Os1	C5	C6	C16	-130.6(4)
Os1	C6	C16	C17	173.7(3)
Os1	C6	C16	C18	-63.6(6)
C1	C2	C3	Os1	-58.7(3)

Atom	Atom	Atom	Atom	Angle/°
C1	C2	C3	C4	-7.1(6)
C1	C2	C7	C8	-97.7(5)
C1	C2	C7	C15	84.0(5)
C1	C6	C16	C17	86.5(4)
C1	C6	C16	C18	-150.8(4)
C2	C1	C6	Os1	50.4(3)
C2	C1	C6	C5	-0.3(5)
C2	C1	C6	C16	-179.7(4)
C2	C3	C4	Os1	-50.1(3)
C2	C3	C4	C5	4.0(6)
C2	C7	C8	C9	-178.8(4)
C2	C7	C15	N1	-1.1(5)
C2	C7	C15	C11	179.1(3)
C3	C2	C7	C8	96.5(5)
C3	C2	C7	C15	-81.8(5)
C3	C4	C5	Os1	-53.2(3)
C3	C4	C5	C6	1.0(6)
C4	C5	C6	Os1	-52.9(3)
C4	C5	C6	C1	-2.9(5)
C4	C5	C6	C16	176.5(3)
C5	C6	C16	C17	-92.9(4)
C5	C6	C16	C18	29.8(6)
C6	C1	C2	Os1	-53.1(3)
C6	C1	C2	C3	5.3(5)
C6	C1	C2	C7	-160.6(3)
C7	C2	C3	Os1	107.1(3)
C7	C2	C3	C4	158.7(4)
C7	C8	C9	C10	0.0(7)
C8	C7	C15	N1	-179.5(4)
C8	C7	C15	C11	0.7(6)
C8	C9	C10	C11	0.4(7)
C9	C10	C11	C12	-179.6(4)
C9	C10	C11	C15	-0.3(6)
C10	C11	C12	C13	-179.2(4)
C10	C11	C15	N1	179.9(4)
C10	C11	C15	C7	-0.3(6)
C11	C12	C13	C14	-1.0(6)
C12	C11	C15	N1	-0.7(6)
C12	C11	C15	C7	179.1(4)
C12	C13	C14	N1	-0.4(7)
C14	N1	C15	C7	179.6(4)
C14	N1	C15	C11	-0.6(5)
C15	N1	C14	C13	1.2(6)
C15	C7	C8	C9	-0.5(6)
C15	C11	C12	C13	1.5(6)

**Table 6:** Hydrogen Fractional Atomic Coordinates ( $\times 10^4$ ) and Equivalent Isotropic Displacement Parameters ( $\text{\AA}^2 \times 10^3$ ) for **04151**.  $U_{eq}$  is defined as 1/3 of the trace of the orthogonalised  $U_{ij}$ .

Atom	x	y	z	$U_{eq}$
H1	4921.28	5561.46	7001.56	26
H3	7938.52	7041.28	9140.2	28
H4	7465.79	7983.13	7246.21	30
H5	5715.29	7740.54	5263.66	27
H8	6236.43	5531.72	10728.42	35
H9	7315.84	4395.2	11958.53	41
H10	8828.21	3647.13	11075.18	38
H12	10080.97	3418.91	9109.41	34
H13	10516.05	3870.23	7006.45	37
H14	9409.22	5021.9	5881.12	31
H16	3830.83	5873.21	4605.85	30
H17A	2621.9	6522.83	6079.64	53

Atom	x	y	z	$U_{eq}$
H17B	1911.64	6709.82	4473.48	53
H17C	2815.56	7391.54	5403.03	53
H18A	4070.1	7452.15	3434.95	55
H18B	3269.25	6698.66	2606.98	55
H18C	4853.79	6664.36	3087.97	55

## Citations

O.V. Dolomanov and L.J. Bourhis and R.J. Gildea and J.A.K. Howard and H. Puschmann, Olex2: A complete structure solution, refinement and analysis program, *J. Appl. Cryst.*, (2009), **42**, 339-341.