

New Stable Aryl-Substituted Acyclic Imino-*N*-Heterocyclic Carbene: Synthesis, Characterisation and Coordination to Early Transition Metals

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Table 1. Crystal data and structure refinement for **2**.

Identification code	k09214	
Empirical formula	C ₂₅ H ₃₁ N ₃	
Formula weight	373.53	
Temperature	150(1) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 8.5723(3) Å	α = 70.656(2)°.
	b = 11.8829(7) Å	β = 88.378(3)°.
	c = 12.3644(8) Å	γ = 69.372(3)°.
Volume	1106.61(10) Å ³	
Z	2	
Density (calculated)	1.121 Mg/m ³	
Absorption coefficient	0.066 mm ⁻¹	
F(000)	404	
Crystal size	0.34 x 0.34 x 0.20 mm ³	
Theta range for data collection	2.59 to 27.50°.	
Index ranges	-11 ≤ h ≤ 11, -14 ≤ k ≤ 15, -15 ≤ l ≤ 15	
Reflections collected	10345	
Independent reflections	4990 [R(int) = 0.0480]	
Completeness to theta = 27.50°	98.1 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.985 and 0.768	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	4990 / 0 / 261	
Goodness-of-fit on F ²	1.049	
Final R indices [I > 2σ(I)]	R1 = 0.0699, wR2 = 0.1747	
R indices (all data)	R1 = 0.1269, wR2 = 0.2146	
Largest diff. peak and hole	0.423 and -0.270 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **2**. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
C(1)	7112(2)	2549(2)	7943(2)	36(1)
C(2)	7055(3)	3033(2)	5977(2)	41(1)
C(3)	6784(3)	4120(2)	6183(2)	41(1)
C(4)	7487(2)	776(2)	7198(2)	32(1)
C(5)	8993(2)	-265(2)	8010(2)	39(1)
C(6)	5014(2)	1388(2)	5985(2)	33(1)
C(7)	4952(3)	1623(2)	4794(2)	38(1)
C(8)	3450(3)	2388(2)	4130(2)	45(1)
C(9)	2029(3)	2912(2)	4618(2)	49(1)
C(10)	2100(3)	2662(2)	5796(2)	44(1)
C(11)	3591(2)	1894(2)	6504(2)	35(1)
C(12)	6499(3)	1020(2)	4265(2)	48(1)
C(13)	3643(3)	1581(2)	7785(2)	41(1)
C(14)	6493(2)	4720(2)	7962(2)	31(1)
C(15)	4832(2)	5442(2)	8040(2)	35(1)
C(16)	4559(2)	6313(2)	8612(2)	36(1)
C(17)	5862(2)	6491(2)	9087(2)	36(1)
C(18)	7497(2)	5739(2)	9003(2)	34(1)
C(19)	7839(2)	4853(2)	8444(2)	33(1)
C(20)	3381(3)	5272(2)	7534(2)	48(1)
C(21)	5511(3)	7498(2)	9640(2)	48(1)
C(22)	9612(2)	4078(2)	8340(2)	41(1)
C(23)	8561(3)	-484(2)	9257(2)	50(1)
C(24)	10518(3)	143(2)	7831(3)	52(1)
C(25)	9374(3)	-1512(2)	7758(3)	56(1)
N(1)	7246(2)	2088(2)	7042(2)	33(1)
N(2)	6813(2)	3805(2)	7374(2)	34(1)
N(3)	6513(2)	482(2)	6685(2)	35(1)

Table 3. Bond lengths [Å] and angles [°] for **2**.

C(1)-N(2)	1.354(3)
C(1)-N(1)	1.380(3)
C(2)-C(3)	1.338(3)
C(2)-N(1)	1.388(3)
C(3)-N(2)	1.393(3)
C(4)-N(3)	1.266(3)
C(4)-N(1)	1.444(3)
C(4)-C(5)	1.526(3)
C(5)-C(24)	1.533(3)
C(5)-C(23)	1.534(3)
C(5)-C(25)	1.535(3)
C(6)-C(11)	1.399(3)
C(6)-C(7)	1.404(3)
C(6)-N(3)	1.425(3)
C(7)-C(8)	1.377(3)
C(7)-C(12)	1.513(3)
C(8)-C(9)	1.382(3)
C(9)-C(10)	1.385(3)
C(10)-C(11)	1.395(3)
C(11)-C(13)	1.500(3)
C(14)-C(19)	1.393(3)
C(14)-C(15)	1.400(3)
C(14)-N(2)	1.445(2)
C(15)-C(16)	1.390(3)
C(15)-C(20)	1.511(3)
C(16)-C(17)	1.387(3)
C(17)-C(18)	1.394(3)
C(17)-C(21)	1.505(3)
C(18)-C(19)	1.388(3)
C(19)-C(22)	1.502(3)
N(2)-C(1)-N(1)	101.48(19)
C(3)-C(2)-N(1)	106.5(2)
C(2)-C(3)-N(2)	106.1(2)

N(3)-C(4)-N(1)	122.38(18)
N(3)-C(4)-C(5)	120.47(18)
N(1)-C(4)-C(5)	117.15(17)
C(4)-C(5)-C(24)	109.91(19)
C(4)-C(5)-C(23)	109.01(16)
C(24)-C(5)-C(23)	110.11(19)
C(4)-C(5)-C(25)	108.90(17)
C(24)-C(5)-C(25)	109.93(18)
C(23)-C(5)-C(25)	109.0(2)
C(11)-C(6)-C(7)	121.56(18)
C(11)-C(6)-N(3)	119.69(19)
C(7)-C(6)-N(3)	118.26(17)
C(8)-C(7)-C(6)	118.5(2)
C(8)-C(7)-C(12)	121.3(2)
C(6)-C(7)-C(12)	120.24(19)
C(7)-C(8)-C(9)	121.2(2)
C(8)-C(9)-C(10)	119.9(2)
C(9)-C(10)-C(11)	121.1(2)
C(10)-C(11)-C(6)	117.8(2)
C(10)-C(11)-C(13)	120.84(18)
C(6)-C(11)-C(13)	121.32(18)
C(19)-C(14)-C(15)	121.82(18)
C(19)-C(14)-N(2)	119.37(16)
C(15)-C(14)-N(2)	118.80(17)
C(16)-C(15)-C(14)	117.67(18)
C(16)-C(15)-C(20)	120.91(18)
C(14)-C(15)-C(20)	121.41(19)
C(17)-C(16)-C(15)	122.38(18)
C(16)-C(17)-C(18)	117.98(19)
C(16)-C(17)-C(21)	120.65(18)
C(18)-C(17)-C(21)	121.34(19)
C(19)-C(18)-C(17)	121.95(19)
C(18)-C(19)-C(14)	118.18(17)
C(18)-C(19)-C(22)	120.88(18)
C(14)-C(19)-C(22)	120.93(18)
C(1)-N(1)-C(2)	112.55(18)

C(1)-N(1)-C(4)	123.25(18)
C(2)-N(1)-C(4)	124.15(17)
C(1)-N(2)-C(3)	113.41(17)
C(1)-N(2)-C(14)	122.37(18)
C(3)-N(2)-C(14)	124.18(18)
C(4)-N(3)-C(6)	123.86(18)

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **2**. The anisotropic displacement factor exponent takes the form: $-2p^2 [h^2 a^* 2 U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
C(1)	35(1)	35(1)	38(1)	-15(1)	3(1)	-10(1)
C(2)	56(1)	40(1)	32(1)	-13(1)	5(1)	-22(1)
C(3)	57(1)	37(1)	31(1)	-7(1)	0(1)	-21(1)
C(4)	34(1)	31(1)	34(1)	-15(1)	11(1)	-13(1)
C(5)	34(1)	31(1)	47(2)	-12(1)	-2(1)	-8(1)
C(6)	40(1)	29(1)	34(1)	-13(1)	1(1)	-14(1)
C(7)	51(1)	34(1)	33(1)	-10(1)	2(1)	-21(1)
C(8)	59(2)	42(1)	36(1)	-8(1)	-3(1)	-26(1)
C(9)	49(1)	42(2)	49(2)	-5(1)	-13(1)	-17(1)
C(10)	39(1)	38(1)	51(2)	-13(1)	0(1)	-11(1)
C(11)	35(1)	33(1)	39(1)	-14(1)	3(1)	-13(1)
C(12)	66(2)	47(2)	39(2)	-22(1)	14(1)	-26(1)
C(13)	38(1)	46(1)	40(1)	-19(1)	7(1)	-14(1)
C(14)	35(1)	25(1)	30(1)	-8(1)	3(1)	-9(1)
C(15)	34(1)	30(1)	37(1)	-8(1)	2(1)	-11(1)
C(16)	33(1)	31(1)	39(1)	-10(1)	7(1)	-8(1)
C(17)	45(1)	32(1)	31(1)	-11(1)	8(1)	-14(1)
C(18)	40(1)	34(1)	30(1)	-9(1)	2(1)	-15(1)
C(19)	34(1)	28(1)	32(1)	-7(1)	1(1)	-10(1)
C(20)	35(1)	44(2)	66(2)	-18(1)	0(1)	-13(1)
C(21)	57(1)	43(1)	50(2)	-25(1)	11(1)	-17(1)
C(22)	36(1)	41(1)	46(2)	-17(1)	0(1)	-11(1)
C(23)	59(2)	40(1)	43(2)	-3(1)	-11(1)	-18(1)
C(24)	33(1)	43(2)	78(2)	-21(1)	-2(1)	-10(1)
C(25)	42(1)	39(2)	83(2)	-25(2)	-6(1)	-5(1)
N(1)	35(1)	32(1)	35(1)	-14(1)	3(1)	-13(1)
N(2)	36(1)	30(1)	34(1)	-12(1)	1(1)	-11(1)
N(3)	36(1)	34(1)	37(1)	-15(1)	5(1)	-12(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **2**.

	x	y	z	U(eq)
H(2)	7107	2929	5246	50
H(3)	6608	4941	5629	50
H(8)	3391	2558	3323	54
H(9)	1006	3443	4147	59
H(10)	1116	3019	6127	53
H(12A)	6185	1129	3471	72
H(12B)	7009	105	4715	72
H(12C)	7303	1435	4268	72
H(13A)	2519	1978	7990	61
H(13B)	4416	1909	8035	61
H(13C)	4028	650	8167	61
H(16)	3439	6804	8680	43
H(18)	8403	5835	9340	41
H(20A)	3414	5490	6699	73
H(20B)	2325	5836	7696	73
H(20C)	3463	4380	7880	73
H(21A)	6514	7329	10120	72
H(21B)	4585	7476	10120	72
H(21C)	5205	8343	9040	72
H(22A)	9803	3165	8691	62
H(22B)	10384	4290	8737	62
H(22C)	9804	4275	7525	62
H(23A)	8228	325	9406	75
H(23B)	9544	-1116	9785	75
H(23C)	7635	-801	9377	75
H(24A)	10697	385	7012	78
H(24B)	11512	-572	8281	78
H(24C)	10319	875	8086	78
H(25A)	8386	-1756	7857	84
H(25B)	10318	-2193	8292	84

H(25C)

9663

-1382

6965

84

Table 6. Crystal data and structure refinement for **3a**.

Identification code	k1010	
Empirical formula	C ₂₆ H ₃₃ Cl ₆ N ₃ Ti	
Formula weight	648.15	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 2 ₁ /n	
Unit cell dimensions	a = 9.7534(5) Å	α = 90°.
	b = 18.8425(11) Å	β = 93.221(3)°.
	c = 16.5548(7) Å	γ = 90°.
Volume	3037.6(3) Å ³	
Z	4	
Density (calculated)	1.417 Mg/m ³	
Absorption coefficient	0.830 mm ⁻¹	
F(000)	1336	
Crystal size	0.15 x 0.06 x 0.04 mm ³	
Theta range for data collection	2.60 to 25.07°.	
Index ranges	-11 ≤ h ≤ 11, -20 ≤ k ≤ 22, -19 ≤ l ≤ 19	
Reflections collected	20804	
Independent reflections	5388 [R(int) = 0.0855]	
Completeness to theta = 25.07°	99.6 %	
Max. and min. transmission	0.9675 and 0.8856	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	5388 / 18 / 337	
Goodness-of-fit on F ²	1.020	
Final R indices [I > 2σ(I)]	R1 = 0.0674, wR2 = 0.1630	
R indices (all data)	R1 = 0.1172, wR2 = 0.1886	
Largest diff. peak and hole	1.290 and -1.080 e.Å ⁻³	

Table 7. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **3a**. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ti(1)	7363(1)	2869(1)	2762(1)	36(1)
Cl(3)	9684(2)	3011(1)	2884(1)	48(1)
Cl(2)	5030(2)	3034(1)	2697(1)	51(1)
Cl(4)	7361(2)	1968(1)	1841(1)	54(1)
Cl(1)	7322(2)	2316(1)	3956(1)	45(1)
N(1)	7457(4)	4496(2)	2683(2)	38(1)
N(2)	7375(4)	4260(2)	3951(3)	39(1)
N(3)	7444(4)	3675(2)	1676(2)	34(1)
C(1)	7395(5)	3945(3)	3223(3)	36(1)
C(2)	7502(6)	5142(3)	3092(4)	48(2)
C(3)	7453(6)	4987(3)	3873(4)	50(2)
C(4)	7493(5)	4345(3)	1827(3)	36(1)
C(5)	7563(6)	5000(3)	1280(3)	41(1)
C(24)	6262(6)	5461(3)	1379(4)	54(2)
C(23)	8918(6)	5406(3)	1503(4)	54(2)
C(25)	7580(7)	4841(3)	367(3)	59(2)
C(14)	7312(6)	3931(3)	4742(3)	39(1)
C(19)	6029(6)	3838(3)	5053(3)	43(1)
C(18)	5997(6)	3578(3)	5846(3)	48(2)
C(17)	7201(7)	3415(3)	6306(3)	50(2)
C(16)	8441(6)	3509(3)	5960(3)	43(1)
C(15)	8537(6)	3773(3)	5182(3)	41(1)
C(22)	4719(6)	4012(3)	4573(4)	56(2)
C(21)	7139(8)	3159(4)	7168(3)	65(2)
C(20)	9916(6)	3900(3)	4845(4)	52(2)
C(6)	7461(6)	3394(3)	853(3)	37(1)
C(11)	6202(6)	3257(3)	430(3)	42(1)
C(10)	6229(7)	2928(3)	-323(3)	50(2)
C(9)	7448(7)	2741(3)	-644(3)	54(2)
C(8)	8660(7)	2893(3)	-226(3)	50(2)
C(7)	8709(6)	3229(3)	533(3)	41(1)

C(13)	4833(6)	3474(4)	733(3)	53(2)
C(12)	10080(6)	3409(4)	928(3)	52(2)
Cl(5)	1708(3)	4732(2)	3309(2)	134(1)
Cl(6)	2686(2)	4958(2)	1730(1)	114(1)
C(1S)	2491(14)	4371(6)	2511(4)	85(4)
C(2S)	3110(10)	4816(9)	2735(3)	85(4)

Table 8. Bond lengths [Å] and angles [°] for **3a**.

Ti(1)-C(1)	2.167(5)
Ti(1)-Cl(1)	2.2370(16)
Ti(1)-Cl(3)	2.2775(18)
Ti(1)-Cl(4)	2.2808(16)
Ti(1)-Cl(2)	2.2930(18)
Ti(1)-N(3)	2.358(4)
N(1)-C(1)	1.373(6)
N(1)-C(2)	1.392(7)
N(1)-C(4)	1.448(6)
N(2)-C(1)	1.344(6)
N(2)-C(3)	1.379(7)
N(2)-C(14)	1.453(7)
N(3)-C(4)	1.288(6)
N(3)-C(6)	1.463(6)
C(2)-C(3)	1.329(8)
C(4)-C(5)	1.535(7)
C(5)-C(25)	1.542(8)
C(5)-C(23)	1.552(8)
C(5)-C(24)	1.554(8)
C(14)-C(19)	1.390(8)
C(14)-C(15)	1.396(8)
C(19)-C(18)	1.403(8)
C(19)-C(22)	1.502(8)
C(18)-C(17)	1.397(8)
C(17)-C(16)	1.379(8)
C(17)-C(21)	1.512(8)
C(16)-C(15)	1.388(7)
C(15)-C(20)	1.504(8)
C(6)-C(7)	1.390(8)
C(6)-C(11)	1.403(7)
C(11)-C(10)	1.394(8)
C(11)-C(13)	1.508(8)
C(10)-C(9)	1.375(9)
C(9)-C(8)	1.367(8)

C(8)-C(7)	1.405(8)
C(7)-C(12)	1.494(8)
Cl(5)-C(1S)	1.704(6)
Cl(5)-C(2S)	1.717(6)
Cl(6)-C(2S)	1.712(6)
Cl(6)-C(1S)	1.721(6)
C(1)-Ti(1)-Cl(1)	97.19(14)
C(1)-Ti(1)-Cl(3)	82.16(15)
Cl(1)-Ti(1)-Cl(3)	92.49(6)
C(1)-Ti(1)-Cl(4)	158.65(15)
Cl(1)-Ti(1)-Cl(4)	104.16(6)
Cl(3)-Ti(1)-Cl(4)	96.34(7)
C(1)-Ti(1)-Cl(2)	83.34(15)
Cl(1)-Ti(1)-Cl(2)	92.17(6)
Cl(3)-Ti(1)-Cl(2)	165.21(7)
Cl(4)-Ti(1)-Cl(2)	96.11(7)
C(1)-Ti(1)-N(3)	70.42(16)
Cl(1)-Ti(1)-N(3)	167.58(11)
Cl(3)-Ti(1)-N(3)	85.17(11)
Cl(4)-Ti(1)-N(3)	88.23(11)
Cl(2)-Ti(1)-N(3)	87.27(11)
C(1)-N(1)-C(2)	110.2(4)
C(1)-N(1)-C(4)	119.6(4)
C(2)-N(1)-C(4)	130.2(4)
C(1)-N(2)-C(3)	110.5(4)
C(1)-N(2)-C(14)	128.6(4)
C(3)-N(2)-C(14)	120.8(4)
C(4)-N(3)-C(6)	122.3(4)
C(4)-N(3)-Ti(1)	119.1(3)
C(6)-N(3)-Ti(1)	118.6(3)
N(2)-C(1)-N(1)	104.7(4)
N(2)-C(1)-Ti(1)	136.7(4)
N(1)-C(1)-Ti(1)	118.6(3)
C(3)-C(2)-N(1)	106.2(5)
C(2)-C(3)-N(2)	108.3(5)

N(3)-C(4)-N(1)	112.3(4)
N(3)-C(4)-C(5)	132.6(5)
N(1)-C(4)-C(5)	115.1(4)
C(4)-C(5)-C(25)	115.2(4)
C(4)-C(5)-C(23)	108.7(4)
C(25)-C(5)-C(23)	105.9(5)
C(4)-C(5)-C(24)	108.9(5)
C(25)-C(5)-C(24)	105.4(5)
C(23)-C(5)-C(24)	112.9(5)
C(19)-C(14)-C(15)	122.8(5)
C(19)-C(14)-N(2)	118.1(5)
C(15)-C(14)-N(2)	118.9(5)
C(14)-C(19)-C(18)	117.2(5)
C(14)-C(19)-C(22)	122.2(5)
C(18)-C(19)-C(22)	120.5(5)
C(17)-C(18)-C(19)	121.6(5)
C(16)-C(17)-C(18)	118.4(5)
C(16)-C(17)-C(21)	121.1(6)
C(18)-C(17)-C(21)	120.5(6)
C(17)-C(16)-C(15)	122.5(5)
C(16)-C(15)-C(14)	117.4(5)
C(16)-C(15)-C(20)	120.6(5)
C(14)-C(15)-C(20)	121.9(5)
C(7)-C(6)-C(11)	121.9(5)
C(7)-C(6)-N(3)	119.5(5)
C(11)-C(6)-N(3)	118.4(5)
C(10)-C(11)-C(6)	117.9(5)
C(10)-C(11)-C(13)	118.7(5)
C(6)-C(11)-C(13)	123.4(5)
C(9)-C(10)-C(11)	121.3(6)
C(8)-C(9)-C(10)	119.5(6)
C(9)-C(8)-C(7)	122.1(6)
C(6)-C(7)-C(8)	117.1(5)
C(6)-C(7)-C(12)	124.3(5)
C(8)-C(7)-C(12)	118.6(5)
C(1S)-Cl(5)-C(2S)	37.0(6)

C(2S)-Cl(6)-C(1S)	36.9(6)
Cl(5)-C(1S)-Cl(6)	113.5(6)
Cl(6)-C(2S)-Cl(5)	113.3(6)

Symmetry transformations used to generate equivalent atoms:

Table 9. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **3a**. The anisotropic displacement factor exponent takes the form: $-2p^2 [h^2 a^* 2 U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
Ti(1)	50(1)	26(1)	34(1)	0(1)	4(1)	0(1)
Cl(3)	50(1)	52(1)	44(1)	7(1)	6(1)	7(1)
Cl(2)	48(1)	51(1)	53(1)	2(1)	1(1)	-6(1)
Cl(4)	92(1)	28(1)	40(1)	-2(1)	7(1)	0(1)
Cl(1)	62(1)	36(1)	38(1)	5(1)	6(1)	0(1)
N(1)	51(3)	25(2)	37(2)	1(2)	2(2)	1(2)
N(2)	45(3)	34(3)	38(2)	-5(2)	6(2)	-1(2)
N(3)	36(2)	33(2)	32(2)	1(2)	2(2)	2(2)
C(1)	43(3)	28(3)	36(3)	-2(2)	1(2)	2(2)
C(2)	71(4)	24(3)	50(4)	-8(3)	5(3)	-4(3)
C(3)	68(4)	30(3)	51(4)	-10(3)	7(3)	-2(3)
C(4)	40(3)	34(3)	35(3)	3(2)	4(2)	2(2)
C(5)	51(3)	30(3)	44(3)	9(2)	5(3)	-1(3)
C(24)	64(4)	32(3)	67(4)	8(3)	4(3)	3(3)
C(23)	56(4)	34(3)	72(4)	6(3)	11(3)	2(3)
C(25)	85(5)	42(4)	49(4)	18(3)	6(3)	0(3)
C(14)	52(4)	30(3)	35(3)	-8(2)	10(3)	0(3)
C(19)	52(4)	37(3)	41(3)	-8(2)	6(3)	2(3)
C(18)	50(4)	46(4)	50(4)	-5(3)	16(3)	-6(3)
C(17)	69(4)	38(3)	42(3)	-1(3)	7(3)	-3(3)
C(16)	47(4)	41(3)	41(3)	-4(3)	0(3)	0(3)
C(15)	45(3)	36(3)	42(3)	-9(2)	7(3)	-3(3)
C(22)	52(4)	60(4)	56(4)	-7(3)	4(3)	8(3)
C(21)	83(5)	68(5)	43(4)	5(3)	10(3)	-10(4)
C(20)	53(4)	52(4)	51(4)	-4(3)	5(3)	-6(3)
C(6)	52(4)	26(3)	34(3)	6(2)	6(3)	3(2)
C(11)	54(4)	34(3)	37(3)	2(2)	0(3)	-2(3)
C(10)	65(4)	50(4)	34(3)	2(3)	-5(3)	-3(3)
C(9)	74(5)	54(4)	32(3)	1(3)	1(3)	0(3)
C(8)	66(4)	50(4)	35(3)	4(3)	12(3)	11(3)
C(7)	53(4)	37(3)	33(3)	6(2)	5(3)	5(3)

C(13)	48(4)	64(4)	46(3)	5(3)	-4(3)	1(3)
C(12)	49(4)	66(4)	41(3)	7(3)	6(3)	1(3)
Cl(5)	117(2)	186(3)	100(2)	36(2)	31(2)	-29(2)
Cl(6)	71(1)	197(3)	76(1)	4(2)	17(1)	0(2)
C(1S)	101(8)	62(7)	87(7)	-21(6)	-44(6)	27(6)
C(2S)	101(8)	62(7)	87(7)	-21(6)	-44(6)	27(6)

Table 10. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **3a**.

	x	y	z	U(eq)
H(2A)	7556	5601	2860	58
H(3A)	7469	5320	4304	59
H(24A)	5444	5193	1193	81
H(24B)	6323	5895	1056	81
H(24C)	6198	5587	1950	81
H(23A)	9692	5147	1290	80
H(23B)	9048	5443	2092	80
H(23C)	8868	5882	1266	80
H(25A)	8424	4585	256	88
H(25B)	7543	5288	63	88
H(25C)	6782	4549	200	88
H(18A)	5136	3510	6075	58
H(16A)	9262	3389	6265	52
H(22A)	4725	3786	4040	84
H(22B)	4641	4527	4507	84
H(22C)	3936	3835	4860	84
H(21A)	7865	3389	7507	97
H(21B)	7270	2643	7186	97
H(21C)	6242	3278	7370	97
H(20A)	10642	3746	5239	78
H(20B)	10024	4407	4733	78
H(20C)	9980	3630	4342	78
H(10A)	5388	2832	-621	60
H(9A)	7446	2507	-1152	64
H(8A)	9498	2768	-456	60
H(13A)	4192	3579	271	80
H(13B)	4952	3898	1073	80
H(13C)	4467	3086	1051	80
H(12A)	9989	3822	1282	77
H(12B)	10723	3521	512	77

H(12C)	10427	3004	1249	77
H(1SA)	1944	3960	2304	102
H(1SB)	3407	4192	2702	102
H(2SA)	3680	5217	2946	102
H(2SB)	3671	4379	2791	102

Table 11. Crystal data and structure refinement for **3b**.

Identification code	k10226	
Empirical formula	C ₂₉ H ₃₉ Cl ₃ N ₃ O Ti	
Formula weight	599.88	
Temperature	150(1) K	
Wavelength	0.71073 Å	
Crystal system	Tetragonal	
Space group	I 41/A	
Unit cell dimensions	a = 34.9199(4) Å	α = 90°.
	b = 34.9199(4) Å	β = 90°.
	c = 11.2791(2) Å	γ = 90°.
Volume	13753.7(3) Å ³	
Z	16	
Density (calculated)	1.159 Mg/m ³	
Absorption coefficient	0.505 mm ⁻¹	
F(000)	5040	
Crystal size	0.34 x 0.24 x 0.20 mm	
Theta range for data collection	2.61 to 27.50°.	
Index ranges	-31 ≤ h ≤ 32, -45 ≤ k ≤ 45, -14 ≤ l ≤ 14	
Reflections collected	51252	
Independent reflections	7841 [R(int) = 0.0670]	
Completeness to theta = 27.50°	99.3 %	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	7841 / 0 / 343	
Goodness-of-fit on F ²	1.115	
Final R indices [I > 2σ(I)]	R1 = 0.0615, wR2 = 0.1605	
R indices (all data)	R1 = 0.0899, wR2 = 0.1788	
Largest diff. peak and hole	0.475 and -0.425 e.Å ⁻³	

Table 12. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **3b**. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ti(1)	4071(1)	9100(1)	1095(1)	34(1)
Cl(2)	4214(1)	8603(1)	2523(1)	50(1)
Cl(1)	3727(1)	8708(1)	-170(1)	53(1)
Cl(3)	3969(1)	9647(1)	-92(1)	45(1)
N(2)	4802(1)	8705(1)	-292(2)	33(1)
N(1)	4933(1)	9055(1)	1221(2)	31(1)
N(3)	4510(1)	9445(1)	2147(2)	32(1)
C(14)	4614(1)	8520(1)	-1283(2)	35(1)
O(1)	3580(1)	9238(1)	2146(2)	48(1)
C(6)	4374(1)	9787(1)	2737(3)	37(1)
C(3)	5187(1)	8647(1)	-40(3)	37(1)
C(5)	5202(1)	9443(1)	2913(3)	41(1)
C(4)	4860(1)	9338(1)	2110(2)	32(1)
C(11)	4169(1)	9760(1)	3797(3)	45(1)
C(12)	4678(1)	10175(1)	1080(3)	50(1)
C(2)	5273(1)	8861(1)	917(2)	36(1)
C(19)	4535(1)	8131(1)	-1188(3)	40(1)
C(24)	5293(1)	9076(1)	3624(3)	45(1)
C(18)	4376(1)	7954(1)	-2188(3)	50(1)
C(17)	4291(1)	8153(1)	-3211(3)	52(1)
C(15)	4536(1)	8733(1)	-2283(3)	40(1)
C(7)	4432(1)	10139(1)	2167(3)	45(1)
C(25)	5107(1)	9751(1)	3838(3)	51(1)
C(8)	4268(1)	10465(1)	2661(4)	55(1)
C(23)	5551(1)	9596(1)	2213(3)	44(1)
C(1)	4642(1)	8950(1)	471(2)	33(1)
C(10)	4011(1)	10094(1)	4257(3)	61(1)
C(13)	4132(1)	9387(1)	4458(3)	60(1)
C(9)	4057(1)	10440(1)	3696(4)	66(1)
C(22)	4604(1)	7910(1)	-69(3)	51(1)
C(20)	4628(1)	9153(1)	-2377(3)	53(1)

C(26)	3327(1)	8944(1)	2617(5)	77(1)
C(16)	4370(1)	8543(1)	-3246(3)	47(1)
C(29)	3371(1)	9597(1)	2143(3)	52(1)
C(21)	4118(2)	7957(1)	-4282(4)	83(1)
C(28)	3087(1)	9563(1)	3136(5)	90(2)
C(27)	3017(1)	9143(1)	3250(5)	91(2)

Table 13. Bond lengths [Å] and angles [°] for **3b**.

Ti(1)-O(1)	2.141(2)
Ti(1)-C(1)	2.178(3)
Ti(1)-N(3)	2.282(2)
Ti(1)-Cl(1)	2.3139(9)
Ti(1)-Cl(3)	2.3595(9)
Ti(1)-Cl(2)	2.4202(9)
N(2)-C(1)	1.336(3)
N(2)-C(3)	1.388(4)
N(2)-C(14)	1.448(3)
N(1)-C(1)	1.374(3)
N(1)-C(2)	1.407(3)
N(1)-C(4)	1.431(3)
N(3)-C(4)	1.280(3)
N(3)-C(6)	1.447(3)
C(14)-C(15)	1.379(4)
C(14)-C(19)	1.389(4)
O(1)-C(29)	1.450(4)
O(1)-C(26)	1.455(4)
C(6)-C(11)	1.397(4)
C(6)-C(7)	1.401(5)
C(3)-C(2)	1.347(4)
C(5)-C(25)	1.534(4)
C(5)-C(4)	1.541(4)
C(5)-C(24)	1.544(4)
C(5)-C(23)	1.548(4)
C(11)-C(10)	1.391(5)
C(11)-C(13)	1.505(5)
C(12)-C(7)	1.503(5)
C(19)-C(18)	1.401(4)
C(19)-C(22)	1.499(4)
C(18)-C(17)	1.380(5)
C(17)-C(16)	1.391(5)
C(17)-C(21)	1.515(5)
C(15)-C(16)	1.398(4)

C(15)-C(20)	1.505(4)
C(7)-C(8)	1.391(4)
C(8)-C(9)	1.383(6)
C(10)-C(9)	1.374(6)
C(26)-C(27)	1.472(6)
C(29)-C(28)	1.501(6)
C(28)-C(27)	1.491(7)
O(1)-Ti(1)-C(1)	165.15(10)
O(1)-Ti(1)-N(3)	97.52(9)
C(1)-Ti(1)-N(3)	71.37(9)
O(1)-Ti(1)-Cl(1)	93.40(7)
C(1)-Ti(1)-Cl(1)	97.65(8)
N(3)-Ti(1)-Cl(1)	169.00(7)
O(1)-Ti(1)-Cl(3)	90.65(6)
C(1)-Ti(1)-Cl(3)	98.61(8)
N(3)-Ti(1)-Cl(3)	88.21(6)
Cl(1)-Ti(1)-Cl(3)	92.96(3)
O(1)-Ti(1)-Cl(2)	87.58(7)
C(1)-Ti(1)-Cl(2)	81.58(7)
N(3)-Ti(1)-Cl(2)	83.92(6)
Cl(1)-Ti(1)-Cl(2)	95.31(4)
Cl(3)-Ti(1)-Cl(2)	171.63(4)
C(1)-N(2)-C(3)	111.5(2)
C(1)-N(2)-C(14)	126.3(2)
C(3)-N(2)-C(14)	122.1(2)
C(1)-N(1)-C(2)	110.2(2)
C(1)-N(1)-C(4)	118.9(2)
C(2)-N(1)-C(4)	130.8(2)
C(4)-N(3)-C(6)	124.7(2)
C(4)-N(3)-Ti(1)	118.07(18)
C(6)-N(3)-Ti(1)	117.04(17)
C(15)-C(14)-C(19)	123.4(3)
C(15)-C(14)-N(2)	118.8(2)
C(19)-C(14)-N(2)	117.8(3)
C(29)-O(1)-C(26)	107.8(3)

C(29)-O(1)-Ti(1)	126.60(19)
C(26)-O(1)-Ti(1)	122.0(2)
C(11)-C(6)-C(7)	121.8(3)
C(11)-C(6)-N(3)	120.3(3)
C(7)-C(6)-N(3)	117.7(3)
C(2)-C(3)-N(2)	107.4(2)
C(25)-C(5)-C(4)	113.6(2)
C(25)-C(5)-C(24)	105.8(3)
C(4)-C(5)-C(24)	105.5(2)
C(25)-C(5)-C(23)	106.0(3)
C(4)-C(5)-C(23)	113.0(2)
C(24)-C(5)-C(23)	112.8(3)
N(3)-C(4)-N(1)	113.3(2)
N(3)-C(4)-C(5)	130.6(2)
N(1)-C(4)-C(5)	116.0(2)
C(10)-C(11)-C(6)	117.6(3)
C(10)-C(11)-C(13)	120.5(3)
C(6)-C(11)-C(13)	121.8(3)
C(3)-C(2)-N(1)	106.0(2)
C(14)-C(19)-C(18)	116.6(3)
C(14)-C(19)-C(22)	122.5(3)
C(18)-C(19)-C(22)	120.8(3)
C(17)-C(18)-C(19)	122.4(3)
C(18)-C(17)-C(16)	118.4(3)
C(18)-C(17)-C(21)	121.6(3)
C(16)-C(17)-C(21)	120.0(3)
C(14)-C(15)-C(16)	117.5(3)
C(14)-C(15)-C(20)	122.8(3)
C(16)-C(15)-C(20)	119.7(3)
C(8)-C(7)-C(6)	118.4(3)
C(8)-C(7)-C(12)	119.6(3)
C(6)-C(7)-C(12)	122.0(3)
C(9)-C(8)-C(7)	120.4(4)
N(2)-C(1)-N(1)	104.9(2)
N(2)-C(1)-Ti(1)	138.2(2)
N(1)-C(1)-Ti(1)	114.52(18)

C(9)-C(10)-C(11)	121.5(4)
C(10)-C(9)-C(8)	120.3(3)
O(1)-C(26)-C(27)	106.8(3)
C(17)-C(16)-C(15)	121.6(3)
O(1)-C(29)-C(28)	105.2(3)
C(27)-C(28)-C(29)	104.6(4)
C(26)-C(27)-C(28)	107.6(3)

Table 14. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **3b**. The anisotropic displacement factor exponent takes the form: $-2p^2 [h^2 a^* 2 U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
Ti(1)	30(1)	28(1)	45(1)	1(1)	0(1)	-1(1)
Cl(2)	56(1)	35(1)	60(1)	11(1)	5(1)	4(1)
Cl(1)	47(1)	42(1)	70(1)	-10(1)	-7(1)	-9(1)
Cl(3)	48(1)	38(1)	51(1)	8(1)	-8(1)	-3(1)
N(2)	35(1)	33(1)	31(1)	-7(1)	-2(1)	5(1)
N(1)	29(1)	34(1)	31(1)	-9(1)	-2(1)	5(1)
N(3)	32(1)	30(1)	34(1)	-1(1)	-1(1)	3(1)
C(14)	37(1)	31(1)	36(1)	-10(1)	-4(1)	2(1)
O(1)	35(1)	38(1)	69(2)	7(1)	14(1)	1(1)
C(6)	34(1)	36(1)	43(2)	-9(1)	-4(1)	8(1)
C(3)	37(1)	37(1)	36(1)	-6(1)	-2(1)	5(1)
C(5)	39(2)	48(2)	34(1)	-10(1)	-6(1)	5(1)
C(4)	35(1)	31(1)	30(1)	-3(1)	-2(1)	-2(1)
C(11)	42(2)	55(2)	39(2)	-12(1)	0(1)	11(1)
C(12)	59(2)	38(2)	51(2)	1(1)	-10(2)	-9(1)
C(2)	28(1)	44(2)	36(1)	-6(1)	-2(1)	5(1)
C(19)	40(2)	37(2)	42(2)	-11(1)	-2(1)	6(1)
C(24)	43(2)	55(2)	36(2)	-5(1)	-2(1)	11(1)
C(18)	58(2)	34(2)	57(2)	-9(1)	-4(2)	-4(1)
C(17)	60(2)	54(2)	41(2)	-13(2)	-10(2)	-5(2)
C(15)	43(2)	38(2)	40(2)	-4(1)	-6(1)	-1(1)
C(7)	49(2)	33(1)	53(2)	-11(1)	-15(1)	8(1)
C(25)	44(2)	64(2)	46(2)	-28(2)	-14(1)	12(2)
C(8)	57(2)	36(2)	72(2)	-11(2)	-19(2)	11(1)
C(23)	35(2)	48(2)	49(2)	-12(1)	-7(1)	-5(1)
C(1)	34(1)	33(1)	32(1)	-1(1)	-2(1)	-1(1)
C(10)	47(2)	85(3)	50(2)	-23(2)	-1(2)	24(2)
C(13)	56(2)	80(3)	45(2)	5(2)	9(2)	11(2)
C(9)	57(2)	60(2)	82(3)	-32(2)	-17(2)	27(2)
C(22)	70(2)	35(2)	48(2)	1(1)	-3(2)	3(2)
C(20)	74(2)	44(2)	41(2)	1(1)	-9(2)	-16(2)

C(26)	63(2)	52(2)	115(4)	15(2)	45(2)	-11(2)
C(16)	56(2)	48(2)	37(2)	-1(1)	-10(1)	1(1)
C(29)	40(2)	45(2)	70(2)	2(2)	7(2)	9(1)
C(21)	107(4)	76(3)	65(3)	-22(2)	-30(3)	-16(3)
C(28)	77(3)	82(3)	110(4)	7(3)	33(3)	24(3)
C(27)	57(3)	89(3)	127(4)	5(3)	34(3)	-13(2)

Table 15. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **3b**.

	x	y	z	U(eq)
H(2)	5354	8489	-455	44
H(25A)	4943	10169	1306	74
H(25B)	4622	10412	686	74
H(25C)	4626	9965	553	74
H(3)	5508	8877	1299	43
H(6A)	5498	9127	4167	67
H(6B)	5367	8876	3088	67
H(6C)	5069	8998	4058	67
H(11)	4326	7693	-2161	59
H(8A)	4905	9661	4345	77
H(8B)	5026	9980	3443	77
H(8C)	5330	9804	4305	77
H(22)	4300	10701	2292	66
H(7A)	5500	9852	1948	66
H(7B)	5597	9434	1540	66
H(7C)	5772	9596	2717	66
H(20)	3872	10083	4959	73
H(24A)	4139	9436	5296	91
H(24B)	4341	9221	4246	91
H(24C)	3894	9267	4254	91
H(21)	3946	10659	4015	79
H(15A)	4491	7660	-141	76
H(15B)	4489	8042	587	76
H(15C)	4874	7885	62	76
H(17A)	4462	9295	-1859	79
H(17B)	4590	9237	-3179	79
H(17C)	4889	9195	-2153	79
H(26A)	3466	8777	3154	92
H(26B)	3223	8790	1977	92
H(13)	4311	8682	-3927	57

H(29A)	3241	9635	1392	62
H(29B)	3543	9811	2279	62
H(16A)	4045	8146	-4857	124
H(16B)	3896	7814	-4042	124
H(16C)	4303	7786	-4624	124
H(28A)	3190	9667	3866	108
H(28B)	2851	9697	2945	108
H(27A)	3015	9069	4079	110
H(27B)	2771	9077	2908	110

Table 16. Crystal data and structure refinement for **4a**.

Identification code	k1024a	
Empirical formula	C ₂₅ H ₃₁ Cl ₄ N ₃ Zr	
Formula weight	606.55	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P n m a	
Unit cell dimensions	a = 25.3593(6) Å	α = 90°.
	b = 10.6747(9) Å	β = 90°.
	c = 16.5496(12) Å	γ = 90°.
Volume	4480.0(5) Å ³	
Z	4	
Density (calculated)	0.899 Mg/m ³	
Absorption coefficient	0.495 mm ⁻¹	
F(000)	1240	
Crystal size	0.20 x 0.18 x 0.09 mm ³	
Theta range for data collection	2.59 to 25.08°.	
Index ranges	-30 ≤ h ≤ 30, -12 ≤ k ≤ 12, -19 ≤ l ≤ 19	
Reflections collected	17689	
Independent reflections	4139 [R(int) = 0.0898]	
Completeness to theta = 25.08°	98.0 %	
Max. and min. transmission	0.9568 and 0.9074	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	4139 / 0 / 180	
Goodness-of-fit on F ²	2.491	
Final R indices [I > 2σ(I)]	R1 = 0.2033, wR2 = 0.5724	
R indices (all data)	R1 = 0.2371, wR2 = 0.5922	
Largest diff. peak and hole	2.713 and -1.076 e.Å ⁻³	

Table 17. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **4a**. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
Zr(1)	2669(1)	2500	3107(1)	46(1)
Cl(3)	1954(2)	2500	4058(3)	83(2)
Cl(2)	2783(2)	251(4)	3058(3)	70(1)
Cl(1)	2256(2)	2500	1813(3)	72(2)
N(2)	3811(6)	2500	2004(10)	66(6)
N(3)	3283(6)	2500	4274(8)	41(4)
C(1)	3540(7)	2500	2727(10)	35(4)
C(3)	4362(8)	2500	2192(14)	85(10)
C(4)	3818(7)	2500	4129(11)	41(4)
C(5)	4261(7)	2500	4708(13)	45(5)
C(6)	3587(8)	2500	1212(11)	57(6)
C(7)	3507(5)	1335(15)	862(9)	51(4)
C(8)	3278(6)	1406(19)	61(9)	62(5)
C(9)	3163(8)	2500	-279(12)	47(5)
C(10)	3621(7)	60(20)	1216(11)	88(6)
C(11)	2908(9)	2500	-1146(16)	64(7)
C(12)	3067(7)	2500	5066(12)	43(5)
C(13)	2942(6)	1335(16)	5440(9)	58(4)
C(14)	2679(6)	1357(17)	6143(10)	58(5)
C(15)	2529(10)	2500	6493(16)	76(8)
C(16)	3112(7)	141(15)	5081(9)	62(4)
C(17)	4086(8)	2500	5647(11)	48(5)
C(18)	4608(7)	1263(17)	4666(12)	74(5)
N(1)	3936(5)	2500	3332(9)	41(4)
C(2)	4417(9)	2500	2907(14)	95(11)

Table 18. Bond lengths [Å] and angles [°] for **4a**.

Zr(1)-C(1)	2.297(17)
Zr(1)-Cl(1)	2.385(5)
Zr(1)-Cl(3)	2.401(5)
Zr(1)-Cl(2)	2.419(5)
Zr(1)-Cl(2)#1	2.419(5)
Zr(1)-N(3)	2.479(14)
N(2)-C(1)	1.38(2)
N(2)-C(3)	1.43(3)
N(2)-C(6)	1.43(2)
N(3)-C(4)	1.38(2)
N(3)-C(12)	1.42(3)
C(1)-N(1)	1.42(2)
C(3)-C(2)	1.19(3)
C(4)-N(1)	1.35(2)
C(4)-C(5)	1.48(3)
C(5)-C(18)	1.589(19)
C(5)-C(18)#1	1.589(19)
C(5)-C(17)	1.62(3)
C(6)-C(7)	1.386(18)
C(6)-C(7)#1	1.386(18)
C(7)-C(8)	1.45(2)
C(7)-C(10)	1.51(2)
C(8)-C(9)	1.33(2)
C(9)-C(8)#1	1.33(2)
C(9)-C(11)	1.57(3)
C(12)-C(13)	1.425(18)
C(12)-C(13)#1	1.425(18)
C(13)-C(14)	1.34(2)
C(13)-C(16)	1.47(2)
C(14)-C(15)	1.40(2)
C(15)-C(14)#1	1.40(2)
N(1)-C(2)	1.41(3)

C(1)-Zr(1)-Cl(1)	100.1(4)
C(1)-Zr(1)-Cl(3)	155.0(4)
Cl(1)-Zr(1)-Cl(3)	104.9(2)
C(1)-Zr(1)-Cl(2)	82.85(13)
Cl(1)-Zr(1)-Cl(2)	91.25(11)
Cl(3)-Zr(1)-Cl(2)	96.48(12)
C(1)-Zr(1)-Cl(2)#1	82.85(13)
Cl(1)-Zr(1)-Cl(2)#1	91.25(11)
Cl(3)-Zr(1)-Cl(2)#1	96.48(12)
Cl(2)-Zr(1)-Cl(2)#1	165.7(3)
C(1)-Zr(1)-N(3)	67.0(5)
Cl(1)-Zr(1)-N(3)	167.2(4)
Cl(3)-Zr(1)-N(3)	87.9(4)
Cl(2)-Zr(1)-N(3)	87.22(11)
Cl(2)#1-Zr(1)-N(3)	87.22(11)
C(1)-N(2)-C(3)	107.3(16)
C(1)-N(2)-C(6)	126.8(16)
C(3)-N(2)-C(6)	126.0(18)
C(4)-N(3)-C(12)	122.6(14)
C(4)-N(3)-Zr(1)	118.8(11)
C(12)-N(3)-Zr(1)	118.5(10)
N(2)-C(1)-N(1)	105.1(14)
N(2)-C(1)-Zr(1)	135.7(12)
N(1)-C(1)-Zr(1)	119.2(11)
C(2)-C(3)-N(2)	109.3(19)
N(1)-C(4)-N(3)	112.8(15)
N(1)-C(4)-C(5)	117.7(16)
N(3)-C(4)-C(5)	129.5(16)
C(4)-C(5)-C(18)	113.1(11)
C(4)-C(5)-C(18)#1	113.1(11)
C(18)-C(5)-C(18)#1	112.5(18)
C(4)-C(5)-C(17)	114.6(15)
C(18)-C(5)-C(17)	101.2(12)
C(18)#1-C(5)-C(17)	101.2(12)
C(7)-C(6)-C(7)#1	127.6(17)

C(7)-C(6)-N(2)	116.1(9)
C(7)#1-C(6)-N(2)	116.1(9)
C(6)-C(7)-C(8)	113.1(14)
C(6)-C(7)-C(10)	128.2(14)
C(8)-C(7)-C(10)	118.7(16)
C(9)-C(8)-C(7)	121.5(17)
C(8)-C(9)-C(8)#1	123(2)
C(8)-C(9)-C(11)	118.5(11)
C(8)#1-C(9)-C(11)	118.5(11)
C(13)-C(12)-C(13)#1	122(2)
C(13)-C(12)-N(3)	119.1(10)
C(13)#1-C(12)-N(3)	119.1(10)
C(14)-C(13)-C(12)	118.2(16)
C(14)-C(13)-C(16)	120.8(15)
C(12)-C(13)-C(16)	121.0(14)
C(13)-C(14)-C(15)	120.5(17)
C(14)-C(15)-C(14)#1	121(2)
C(4)-N(1)-C(2)	132.7(17)
C(4)-N(1)-C(1)	122.1(14)
C(2)-N(1)-C(1)	105.1(16)
C(3)-C(2)-N(1)	113(2)

Symmetry transformations used to generate equivalent atoms:

#1 x,-y+1/2,z

Table 19. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **4a**. The anisotropic displacement factor exponent takes the form: $-2p^2 [h^2 a^* 2 U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
Zr(1)	31(1)	72(2)	36(1)	0	-1(1)	0
Cl(3)	36(3)	171(7)	43(3)	0	4(2)	0
Cl(2)	92(3)	65(3)	52(2)	-1(2)	-7(2)	-15(2)
Cl(1)	50(3)	130(6)	38(3)	0	-11(2)	0
N(2)	27(9)	127(18)	42(10)	0	-13(7)	0
N(3)	38(8)	58(10)	26(8)	0	-2(6)	0
C(1)	40(10)	40(11)	26(9)	0	-2(7)	0
C(3)	28(11)	190(30)	39(13)	0	14(9)	0
C(4)	40(10)	43(11)	40(10)	0	-6(8)	0
C(5)	44(10)	40(11)	52(12)	0	5(9)	0
C(6)	57(13)	100(20)	15(9)	0	-11(8)	0
C(7)	30(7)	80(12)	42(8)	12(8)	3(6)	-4(6)
C(8)	45(8)	104(14)	38(8)	2(9)	-1(6)	-12(8)
C(9)	46(11)	58(14)	37(10)	0	-1(8)	0
C(10)	78(12)	135(18)	51(9)	20(12)	-1(9)	43(12)
C(11)	47(12)	59(14)	86(17)	0	-41(12)	0
C(12)	26(8)	57(12)	46(11)	0	-13(8)	0
C(13)	44(8)	81(12)	48(9)	21(8)	-4(7)	-12(7)
C(14)	56(9)	78(13)	41(9)	9(8)	-13(7)	-18(8)
C(15)	55(13)	130(30)	48(14)	0	13(12)	0
C(16)	84(12)	64(11)	39(8)	-1(8)	-11(8)	-2(9)
C(17)	46(11)	66(14)	32(10)	0	-7(8)	0
C(18)	62(10)	76(13)	83(13)	-16(10)	-4(9)	10(8)
N(1)	23(7)	64(11)	36(8)	0	1(6)	0
C(2)	30(11)	220(40)	38(14)	0	17(9)	0

Table 20. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **4a**.

	x	y	z	U(eq)
H(3)	4640	2500	1808	102
H(8)	3208	655	-226	75
H(10A)	3886	142	1643	132
H(10B)	3754	-494	792	132
H(10C)	3296	-288	1444	132
H(11A)	3174	2741	-1547	96
H(11B)	2615	3099	-1161	96
H(11C)	2775	1660	-1271	96
H(14)	2594	592	6406	70
H(15)	2323	2500	6973	91
H(16A)	3146	-493	5506	94
H(16B)	3454	256	4813	94
H(16C)	2851	-137	4683	94
H(17A)	4368	2130	5975	73
H(17B)	3763	2007	5711	73
H(17C)	4022	3363	5824	73
H(18A)	4920	1360	5010	111
H(18B)	4719	1115	4107	111
H(18C)	4399	548	4856	111
H(2)	4751	2500	3165	114

Table 21. Crystal data and structure refinement for **6b**.

Identification code	edwin_0m	
Empirical formula	C ₃₀ H ₄₁ Cl ₅ Cr N ₃ O	
Formula weight	688.91	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Tetragonal	
Space group	I4(1)/a	
Unit cell dimensions	a = 34.553(6) Å	α = 90°.
	b = 34.553 Å	β = 90°.
	c = 11.316(2) Å	γ = 90°.
Volume	13510(3) Å ³	
Z	16	
Density (calculated)	1.355 Mg/m ³	
Absorption coefficient	0.762 mm ⁻¹	
F(000)	5744	
Crystal size	0.349 x 0.101 x 0.083 mm ³	
Theta range for data collection	1.89 to 24.99°.	
Index ranges	-31 ≤ h ≤ 41, -41 ≤ k ≤ 33, -13 ≤ l ≤ 13	
Reflections collected	62831	
Independent reflections	5953 [R(int) = 0.1289]	
Completeness to theta = 24.99°	100.0 %	
Absorption correction	Numerical	
Max. and min. transmission	0.9759 and 0.8123	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	5953 / 12 / 396	
Goodness-of-fit on F ²	1.372	
Final R indices [I > 2σ(I)]	R1 = 0.0595, wR2 = 0.1178	
R indices (all data)	R1 = 0.0942, wR2 = 0.1291	
Largest diff. peak and hole	0.583 and -0.619 e.Å ⁻³	

Table 22. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **6b**. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
Cr(1)	891(1)	899(1)	1174(1)	19(1)
Cl(2)	758(1)	1404(1)	2518(1)	29(1)
Cl(1)	1263(1)	1295(1)	5(1)	29(1)
Cl(3)	979(1)	387(1)	-91(1)	25(1)
O(1)	1378(1)	747(1)	2184(3)	30(1)
N(1)	58(1)	942(1)	1212(3)	17(1)
N(2)	212(1)	1304(1)	-275(3)	17(1)
N(3)	482(1)	555(1)	2156(3)	20(1)
C(1)	364(1)	1052(1)	503(4)	16(1)
C(2)	-280(1)	1140(1)	868(4)	20(1)
C(3)	-182(1)	1360(1)	-60(4)	19(1)
C(4)	124(1)	658(1)	2100(4)	17(1)
C(5)	-219(1)	548(1)	2888(4)	23(1)
C(24)	-314(1)	921(1)	3602(4)	26(1)
C(23)	-571(1)	395(1)	2181(4)	27(1)
C(25)	-124(1)	238(1)	3817(4)	33(1)
C(14)	408(1)	1491(1)	-1258(4)	18(1)
C(19)	488(1)	1887(1)	-1162(4)	21(1)
C(18)	646(1)	2068(1)	-2147(4)	28(1)
C(17)	730(1)	1865(1)	-3184(4)	27(1)
C(16)	649(1)	1472(1)	-3227(4)	28(1)
C(15)	483(1)	1278(1)	-2265(4)	22(1)
C(22)	412(1)	2106(1)	-29(4)	27(1)
C(21)	900(2)	2069(2)	-4242(5)	44(1)
C(20)	381(1)	856(1)	-2372(4)	30(1)
C(6)	624(1)	209(1)	2744(4)	24(1)
C(11)	834(1)	238(1)	3793(4)	30(1)
C(10)	999(1)	-100(2)	4251(5)	42(1)
C(9)	955(1)	-451(2)	3683(5)	46(2)
C(8)	741(1)	-474(1)	2659(5)	38(1)
C(7)	567(1)	-147(1)	2164(4)	28(1)

C(13)	862(2)	610(2)	4474(5)	43(1)
C(12)	315(1)	-186(1)	1094(4)	31(1)
C(26)	1606(1)	397(1)	2009(5)	35(1)
C(27)	1901(2)	406(2)	3003(6)	58(2)
C(28)	1981(2)	837(2)	3124(7)	68(2)
C(29)	1636(2)	1042(1)	2688(6)	59(2)
C(30)	1995(3)	9208(3)	5520(11)	77(3)
Cl(4)	1915(1)	9233(1)	3991(3)	87(1)
Cl(5)	2013(1)	9670(1)	6083(3)	109(1)
C(30A)	1819(10)	9270(11)	5710(30)	77(3)
Cl(4A)	1900(3)	9017(3)	4385(9)	87(1)
Cl(5A)	2274(4)	9570(3)	6126(10)	109(1)

Table 23. Bond lengths [Å] and angles [°] for **6b**.

Cr(1)-C(1)	2.041(4)
Cr(1)-O(1)	2.100(3)
Cr(1)-N(3)	2.156(3)
Cr(1)-Cl(1)	2.2954(13)
Cr(1)-Cl(3)	2.2985(12)
Cr(1)-Cl(2)	2.3596(12)
O(1)-C(26)	1.458(5)
O(1)-C(29)	1.469(5)
N(1)-C(1)	1.380(5)
N(1)-C(2)	1.408(5)
N(1)-C(4)	1.424(5)
N(2)-C(1)	1.345(5)
N(2)-C(3)	1.396(5)
N(2)-C(14)	1.453(5)
N(3)-C(4)	1.287(5)
N(3)-C(6)	1.454(5)
C(2)-C(3)	1.340(6)
C(2)-H(2)	0.9500
C(3)-H(3)	0.9500
C(4)-C(5)	1.533(6)
C(5)-C(25)	1.537(6)
C(5)-C(23)	1.546(6)
C(5)-C(24)	1.555(6)
C(24)-H(6A)	0.9800
C(24)-H(6B)	0.9800
C(24)-H(6C)	0.9800
C(23)-H(7A)	0.9800
C(23)-H(7B)	0.9800
C(23)-H(7C)	0.9800
C(25)-H(8A)	0.9800
C(25)-H(8B)	0.9800
C(25)-H(8C)	0.9800
C(14)-C(15)	1.381(6)
C(14)-C(19)	1.401(5)

C(19)-C(18)	1.388(6)
C(19)-C(22)	1.512(6)
C(18)-C(17)	1.396(6)
C(18)-H(11)	0.9500
C(17)-C(16)	1.387(6)
C(17)-C(21)	1.508(6)
C(16)-C(15)	1.401(6)
C(16)-H(13)	0.9500
C(15)-C(20)	1.506(5)
C(22)-H(15A)	0.9800
C(22)-H(15B)	0.9800
C(22)-H(15C)	0.9800
C(21)-H(16A)	0.9800
C(21)-H(16B)	0.9800
C(21)-H(16C)	0.9800
C(20)-H(17A)	0.9800
C(20)-H(17B)	0.9800
C(20)-H(17C)	0.9800
C(6)-C(11)	1.394(6)
C(6)-C(7)	1.410(6)
C(11)-C(10)	1.397(6)
C(11)-C(13)	1.501(7)
C(10)-C(9)	1.382(8)
C(10)-H(20)	0.9500
C(9)-C(8)	1.375(8)
C(9)-H(21)	0.9500
C(8)-C(7)	1.397(6)
C(8)-H(22)	0.9500
C(7)-C(12)	1.497(7)
C(13)-H(24A)	0.9800
C(13)-H(24B)	0.9800
C(13)-H(24C)	0.9800
C(12)-H(25A)	0.9800
C(12)-H(25B)	0.9800
C(12)-H(25C)	0.9800
C(26)-C(27)	1.520(7)

C(26)-H(26A)	0.9900
C(26)-H(26B)	0.9900
C(27)-C(28)	1.523(8)
C(27)-H(27A)	0.9900
C(27)-H(27B)	0.9900
C(28)-C(29)	1.471(7)
C(28)-H(28A)	0.9900
C(28)-H(28B)	0.9900
C(29)-H(29A)	0.9900
C(29)-H(29B)	0.9900
C(30)-Cl(5)	1.718(13)
C(30)-Cl(4)	1.754(13)
C(30)-H(30A)	0.9900
C(30)-H(30B)	0.9900
C(30A)-Cl(4A)	1.75(4)
C(30A)-Cl(5A)	1.94(4)
C(30A)-H(30C)	0.9900
C(30A)-H(30D)	0.9900
C(1)-Cr(1)-O(1)	168.84(15)
C(1)-Cr(1)-N(3)	75.50(14)
O(1)-Cr(1)-N(3)	96.10(13)
C(1)-Cr(1)-Cl(1)	97.53(11)
O(1)-Cr(1)-Cl(1)	90.83(9)
N(3)-Cr(1)-Cl(1)	173.03(10)
C(1)-Cr(1)-Cl(3)	94.86(11)
O(1)-Cr(1)-Cl(3)	92.28(9)
N(3)-Cr(1)-Cl(3)	89.00(9)
Cl(1)-Cr(1)-Cl(3)	91.48(5)
C(1)-Cr(1)-Cl(2)	82.82(11)
O(1)-Cr(1)-Cl(2)	89.50(9)
N(3)-Cr(1)-Cl(2)	86.99(9)
Cl(1)-Cr(1)-Cl(2)	92.33(5)
Cl(3)-Cr(1)-Cl(2)	175.77(5)
C(26)-O(1)-C(29)	107.4(3)
C(26)-O(1)-Cr(1)	124.6(3)

C(29)-O(1)-Cr(1)	121.6(3)
C(1)-N(1)-C(2)	110.0(3)
C(1)-N(1)-C(4)	118.5(3)
C(2)-N(1)-C(4)	131.5(3)
C(1)-N(2)-C(3)	110.9(3)
C(1)-N(2)-C(14)	127.4(3)
C(3)-N(2)-C(14)	121.6(3)
C(4)-N(3)-C(6)	125.1(3)
C(4)-N(3)-Cr(1)	116.9(3)
C(6)-N(3)-Cr(1)	117.8(2)
N(2)-C(1)-N(1)	105.0(3)
N(2)-C(1)-Cr(1)	139.3(3)
N(1)-C(1)-Cr(1)	113.3(3)
C(3)-C(2)-N(1)	106.3(3)
C(3)-C(2)-H(2)	126.8
N(1)-C(2)-H(2)	126.8
C(2)-C(3)-N(2)	107.8(3)
C(2)-C(3)-H(3)	126.1
N(2)-C(3)-H(3)	126.1
N(3)-C(4)-N(1)	112.3(3)
N(3)-C(4)-C(5)	130.3(4)
N(1)-C(4)-C(5)	117.3(3)
C(4)-C(5)-C(25)	113.9(3)
C(4)-C(5)-C(23)	113.1(3)
C(25)-C(5)-C(23)	106.5(4)
C(4)-C(5)-C(24)	105.0(3)
C(25)-C(5)-C(24)	105.5(3)
C(23)-C(5)-C(24)	112.8(3)
C(5)-C(24)-H(6A)	109.5
C(5)-C(24)-H(6B)	109.5
H(6A)-C(24)-H(6B)	109.5
C(5)-C(24)-H(6C)	109.5
H(6A)-C(24)-H(6C)	109.5
H(6B)-C(24)-H(6C)	109.5
C(5)-C(23)-H(7A)	109.5
C(5)-C(23)-H(7B)	109.5

H(7A)-C(23)-H(7B)	109.5
C(5)-C(23)-H(7C)	109.5
H(7A)-C(23)-H(7C)	109.5
H(7B)-C(23)-H(7C)	109.5
C(5)-C(25)-H(8A)	109.5
C(5)-C(25)-H(8B)	109.5
H(8A)-C(25)-H(8B)	109.5
C(5)-C(25)-H(8C)	109.5
H(8A)-C(25)-H(8C)	109.5
H(8B)-C(25)-H(8C)	109.5
C(15)-C(14)-C(19)	123.2(4)
C(15)-C(14)-N(2)	118.8(3)
C(19)-C(14)-N(2)	117.9(4)
C(18)-C(19)-C(14)	117.0(4)
C(18)-C(19)-C(22)	121.6(4)
C(14)-C(19)-C(22)	121.3(4)
C(19)-C(18)-C(17)	122.1(4)
C(19)-C(18)-H(11)	118.9
C(17)-C(18)-H(11)	118.9
C(16)-C(17)-C(18)	118.5(4)
C(16)-C(17)-C(21)	120.5(4)
C(18)-C(17)-C(21)	121.0(4)
C(17)-C(16)-C(15)	121.6(4)
C(17)-C(16)-H(13)	119.2
C(15)-C(16)-H(13)	119.2
C(14)-C(15)-C(16)	117.6(4)
C(14)-C(15)-C(20)	122.6(4)
C(16)-C(15)-C(20)	119.8(4)
C(19)-C(22)-H(15A)	109.5
C(19)-C(22)-H(15B)	109.5
H(15A)-C(22)-H(15B)	109.5
C(19)-C(22)-H(15C)	109.5
H(15A)-C(22)-H(15C)	109.5
H(15B)-C(22)-H(15C)	109.5
C(17)-C(21)-H(16A)	109.5
C(17)-C(21)-H(16B)	109.5

H(16A)-C(21)-H(16B)	109.5
C(17)-C(21)-H(16C)	109.5
H(16A)-C(21)-H(16C)	109.5
H(16B)-C(21)-H(16C)	109.5
C(15)-C(20)-H(17A)	109.5
C(15)-C(20)-H(17B)	109.5
H(17A)-C(20)-H(17B)	109.5
C(15)-C(20)-H(17C)	109.5
H(17A)-C(20)-H(17C)	109.5
H(17B)-C(20)-H(17C)	109.5
C(11)-C(6)-C(7)	122.1(4)
C(11)-C(6)-N(3)	120.5(4)
C(7)-C(6)-N(3)	117.3(4)
C(6)-C(11)-C(10)	118.0(5)
C(6)-C(11)-C(13)	122.1(4)
C(10)-C(11)-C(13)	119.8(5)
C(9)-C(10)-C(11)	121.0(5)
C(9)-C(10)-H(20)	119.5
C(11)-C(10)-H(20)	119.5
C(8)-C(9)-C(10)	120.2(5)
C(8)-C(9)-H(21)	119.9
C(10)-C(9)-H(21)	119.9
C(9)-C(8)-C(7)	121.5(5)
C(9)-C(8)-H(22)	119.3
C(7)-C(8)-H(22)	119.3
C(8)-C(7)-C(6)	117.3(5)
C(8)-C(7)-C(12)	120.3(4)
C(6)-C(7)-C(12)	122.4(4)
C(11)-C(13)-H(24A)	109.5
C(11)-C(13)-H(24B)	109.5
H(24A)-C(13)-H(24B)	109.5
C(11)-C(13)-H(24C)	109.5
H(24A)-C(13)-H(24C)	109.5
H(24B)-C(13)-H(24C)	109.5
C(7)-C(12)-H(25A)	109.5
C(7)-C(12)-H(25B)	109.5

H(25A)-C(12)-H(25B)	109.5
C(7)-C(12)-H(25C)	109.5
H(25A)-C(12)-H(25C)	109.5
H(25B)-C(12)-H(25C)	109.5
O(1)-C(26)-C(27)	104.3(4)
O(1)-C(26)-H(26A)	110.9
C(27)-C(26)-H(26A)	110.9
O(1)-C(26)-H(26B)	110.9
C(27)-C(26)-H(26B)	110.9
H(26A)-C(26)-H(26B)	108.9
C(28)-C(27)-C(26)	102.0(4)
C(28)-C(27)-H(27A)	111.4
C(26)-C(27)-H(27A)	111.4
C(28)-C(27)-H(27B)	111.4
C(26)-C(27)-H(27B)	111.4
H(27A)-C(27)-H(27B)	109.2
C(29)-C(28)-C(27)	107.0(4)
C(29)-C(28)-H(28A)	110.3
C(27)-C(28)-H(28A)	110.3
C(29)-C(28)-H(28B)	110.3
C(27)-C(28)-H(28B)	110.3
H(28A)-C(28)-H(28B)	108.6
O(1)-C(29)-C(28)	106.9(4)
O(1)-C(29)-H(29A)	110.3
C(28)-C(29)-H(29A)	110.3
O(1)-C(29)-H(29B)	110.3
C(28)-C(29)-H(29B)	110.3
H(29A)-C(29)-H(29B)	108.6
Cl(5)-C(30)-Cl(4)	109.0(7)
Cl(5)-C(30)-H(30A)	109.9
Cl(4)-C(30)-H(30A)	109.9
Cl(5)-C(30)-H(30B)	109.9
Cl(4)-C(30)-H(30B)	109.9
H(30A)-C(30)-H(30B)	108.3
Cl(4A)-C(30A)-Cl(5A)	110.3(18)
Cl(4A)-C(30A)-H(30C)	109.6

Cl(5A)-C(30A)-H(30C)	109.6
Cl(4A)-C(30A)-H(30D)	109.6
Cl(5A)-C(30A)-H(30D)	109.6
H(30C)-C(30A)-H(30D)	108.1

Table 24. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **6b**. The anisotropic displacement factor exponent takes the form: $-2p^2 [h^2 a^* 2 U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
Cr(1)	19(1)	15(1)	24(1)	-2(1)	-3(1)	1(1)
Cl(2)	36(1)	19(1)	31(1)	-7(1)	-5(1)	4(1)
Cl(1)	24(1)	21(1)	41(1)	0(1)	4(1)	-5(1)
Cl(3)	25(1)	21(1)	28(1)	-6(1)	3(1)	-1(1)
O(1)	28(2)	21(2)	41(2)	-6(1)	-12(2)	1(1)
N(1)	21(2)	17(2)	14(2)	6(2)	1(2)	0(1)
N(2)	18(2)	17(2)	16(2)	4(1)	-1(1)	1(1)
N(3)	26(2)	18(2)	14(2)	0(1)	-1(2)	4(2)
C(1)	21(2)	16(2)	12(2)	-4(2)	5(2)	-4(2)
C(2)	17(2)	24(2)	20(2)	2(2)	1(2)	2(2)
C(3)	16(2)	24(2)	18(2)	2(2)	2(2)	2(2)
C(4)	25(2)	12(2)	14(2)	0(2)	-1(2)	2(2)
C(5)	28(2)	28(2)	13(2)	7(2)	5(2)	5(2)
C(24)	32(2)	32(2)	15(2)	5(2)	3(2)	11(2)
C(23)	27(2)	22(2)	33(3)	5(2)	9(2)	-2(2)
C(25)	36(3)	36(3)	27(3)	14(2)	11(2)	11(2)
C(14)	15(2)	20(2)	19(2)	7(2)	3(2)	0(2)
C(19)	17(2)	24(2)	22(2)	6(2)	2(2)	4(2)
C(18)	29(2)	18(2)	36(3)	6(2)	5(2)	0(2)
C(17)	29(2)	27(2)	26(3)	8(2)	8(2)	1(2)
C(16)	31(2)	31(3)	22(3)	-1(2)	5(2)	1(2)
C(15)	24(2)	20(2)	21(2)	-1(2)	3(2)	-1(2)
C(22)	34(2)	23(2)	26(3)	-1(2)	2(2)	3(2)
C(21)	57(3)	39(3)	34(3)	13(2)	20(3)	-2(3)
C(20)	46(3)	24(2)	21(3)	-2(2)	4(2)	-9(2)
C(6)	26(2)	25(2)	22(3)	10(2)	3(2)	7(2)
C(11)	25(2)	41(3)	25(3)	8(2)	4(2)	10(2)
C(10)	38(3)	56(4)	31(3)	23(3)	1(2)	17(3)
C(9)	39(3)	43(3)	56(4)	30(3)	21(3)	21(2)
C(8)	40(3)	25(2)	49(4)	13(2)	21(3)	13(2)
C(7)	28(2)	23(2)	32(3)	8(2)	13(2)	2(2)

C(13)	48(3)	58(3)	24(3)	-4(3)	-7(2)	9(3)
C(12)	40(3)	21(2)	34(3)	0(2)	8(2)	-8(2)
C(26)	26(2)	32(3)	48(3)	-1(2)	-5(2)	12(2)
C(27)	46(3)	53(4)	74(5)	-6(3)	-29(3)	18(3)
C(28)	32(3)	64(4)	109(6)	-30(4)	-24(3)	2(3)
C(29)	49(3)	30(3)	97(5)	-12(3)	-46(3)	-7(2)
C(30)	92(7)	73(5)	67(5)	13(4)	-6(5)	-1(5)
Cl(4)	77(2)	121(3)	62(2)	-13(2)	3(2)	-9(2)
Cl(5)	145(4)	75(2)	107(2)	-27(2)	1(3)	7(2)
C(30A)	92(7)	73(5)	67(5)	13(4)	-6(5)	-1(5)
Cl(4A)	77(2)	121(3)	62(2)	-13(2)	3(2)	-9(2)
Cl(5A)	145(4)	75(2)	107(2)	-27(2)	1(3)	7(2)

Table 25. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **6b**.

	x	y	z	U(eq)
H(2)	-528	1122	1224	24
H(3)	-350	1524	-495	23
H(6A)	-85	999	4055	40
H(6B)	-529	869	4144	40
H(6C)	-386	1128	3056	40
H(7A)	-625	570	1521	41
H(7B)	-797	381	2700	41
H(7C)	-512	136	1874	41
H(8A)	-48	-3	3419	49
H(8B)	-353	189	4307	49
H(8C)	89	328	4317	49
H(11)	699	2337	-2114	33
H(13)	707	1331	-3925	34
H(15A)	531	2363	-77	41
H(15B)	523	1963	638	41
H(15C)	132	2134	85	41
H(16A)	1162	2159	-4051	65
H(16B)	738	2291	-4453	65
H(16C)	913	1889	-4910	65
H(17A)	548	704	-1848	45
H(17B)	419	771	-3190	45
H(17C)	110	817	-2146	45
H(20)	1144	-87	4962	50
H(21)	1072	-677	4000	55
H(22)	712	-718	2281	46
H(24A)	1120	632	4824	65
H(24B)	667	612	5102	65
H(24C)	817	828	3939	65
H(25A)	364	-436	713	47
H(25B)	373	24	539	47

H(25C)	42	-170	1330	47
H(26A)	1735	400	1228	43
H(26B)	1441	163	2064	43
H(27A)	1793	297	3742	69
H(27B)	2139	262	2787	69
H(28A)	2031	904	3961	82
H(28B)	2211	910	2652	82
H(29A)	1506	1179	3345	71
H(29B)	1709	1233	2078	71
H(30A)	1782	9062	5902	93
H(30B)	2241	9072	5682	93
H(30C)	1759	9085	6347	93
H(30D)	1594	9445	5609	93
