Supporting Information

Manuscript name: Addition of Ynamines to Tungsten Vinylidene Complexes (η^5-C_5H_5)(NO)(CO)W=C=C(H)R

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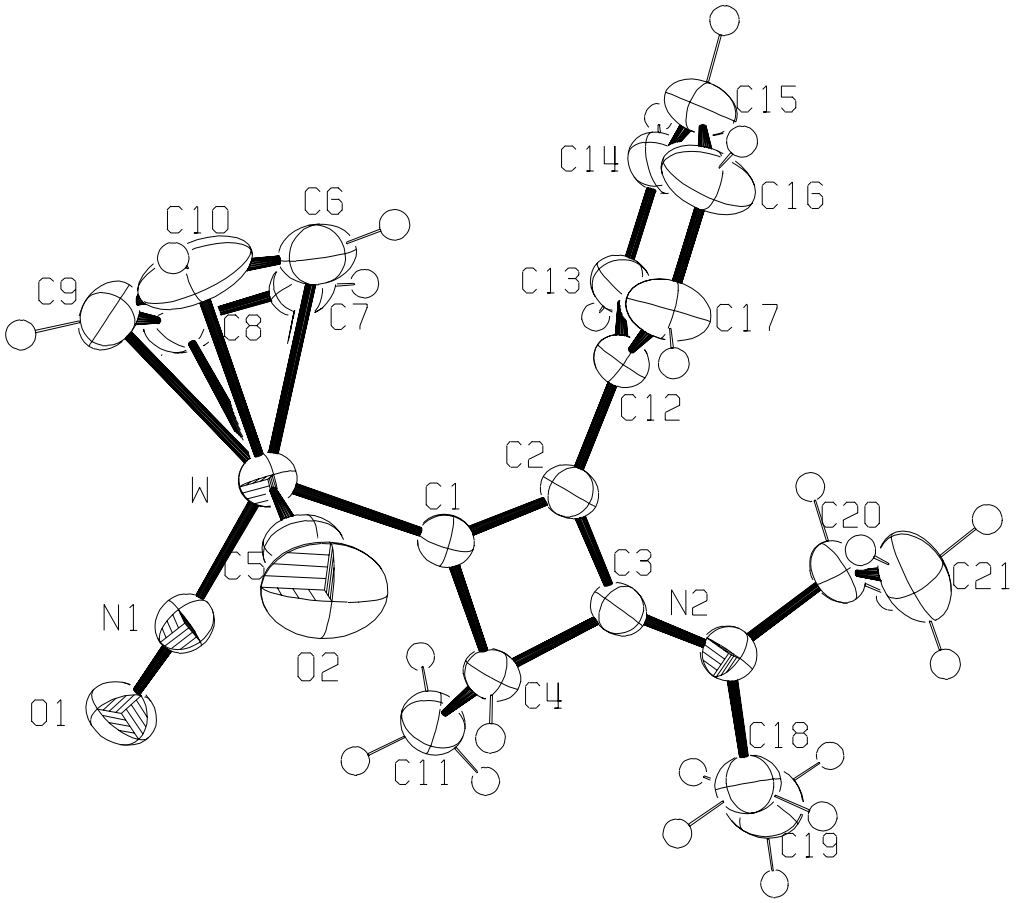

Figure 1. Molecular structure and atom numbering scheme for **4a** with H atoms. Thermal ellipsoids shown at 40%.

Table 1. Crystal Data and Conditions for Crystallographic Data Collection and Structure Refinement for **4a**.

Formula	$C_{21}H_{24}N_2O_2W$
Fw	520.27
Color	red, transparent
cryst syst	monoclinic
space group	$P2_1/n$
cell params	a = 9.471(1) Å, α = 90°
	b = 15.003(2) Å, β = 107.82(1)°
	c = 15.022(2) Å, γ = 90°
V/ Å^3	2032.06
Z	4
d_{calc}/g.cm^{-3}	1.70
μ/cm^{-1}	57
diffractometer	Image Plate Diffractometer system (STOE)
radiation	Mo K_α
monochromator	graphite
2θ/deg	$3.81° \leq 2\theta \leq 56.30°$
no. Of reflns meads	18300
no. of indep rflns	4788
R_{int}	0.0338
reflns with $F_\circ > 4\sigma(F_\circ)$	3697
temperature/K	295
applied corrections	Lorenz and polarization coefficients
structure determination and refinement	W positional params from direct methods (SHELX-97);[a]
	further atoms from ΔF synthesis (SHELX-97),[b] refinement
	by anisotropic full-matrix least-squares procedure for
	all non-hydrogens; hydrogen position refinement by riding
	model; atomic scattering factors from literature.[c]
refined params	235
wR2	0.0598
R1	0.0447
R1 [$F_\circ > 4\sigma(F_\circ)$]	0.027
max and min in $\Delta\sigma$ (e A^{-3})	0.66, -0.60

[a] Sheldrick, G. M. SHELXS-97, program for the Solution of Crystal Structures; Universität Göttingen, 1997.

[b] Sheldrick, G. M. SHELXL-97, Program for Crystal Structure Reifnement; Universität Göttingen, 1997.

[c] *International Tables for Crystalography*; Wilson , A. J. C., Ed.; Kluwer Academic: Dordrecht , The Netherlands, 1992; Vol. C.

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

	x	y	z	U(eq)
W	3547(1)	9729(1)	1453(1)	39(1)
C(6)	1761(6)	10885(3)	965(4)	62(1)
C(7)	2967(6)	11237(3)	1630(4)	54(1)
C(8)	4178(6)	11232(3)	1305(4)	62(1)
C(9)	3741(8)	10868(4)	410(4)	78(2)
C(10)	2229(9)	10646(4)	201(4)	86(2)
C(1)	3133(4)	9410(2)	2673(3)	37(1)
C(2)	2134(4)	9641(3)	3175(3)	39(1)
C(12)	908(4)	10300(3)	2971(3)	39(1)
C(13)	1143(5)	11158(3)	3326(3)	47(1)
C(14)	32(5)	11788(3)	3075(4)	55(1)
C(15)	-1324(6)	11576(3)	2467(4)	63(1)
C(16)	-1596(5)	10720(4)	2133(5)	72(2)
C(17)	-495(5)	10083(3)	2382(4)	57(1)
C(3)	2705(4)	9005(2)	3869(3)	38(1)
N(2)	2386(4)	8713(2)	4611(3)	43(1)
C(18)	3209(5)	7989(3)	5196(3)	50(1)
C(19)	4195(7)	8311(4)	6127(4)	75(2)
C(20)	1078(5)	9058(3)	4818(3)	53(1)
C(21)	-286(6)	8496(5)	4384(5)	82(2)
C(4)	3886(4)	8709(3)	3435(3)	41(1)
C(11)	5497(5)	8888(4)	3970(4)	60(1)
N(1)	5269(4)	9137(2)	1701(3)	44(1)
O(1)	6466(3)	8781(2)	1825(3)	63(1)
O(2)	1727(5)	8061(3)	545(4)	101(2)
C(5)	2404(5)	8663(3)	892(4)	58(1)

Table 3. Bond lengths [Å] and angles [°] for **4a**.

W-N(1)	1.793(3)
W-C(5)	1.970(5)
W-C(1)	2.045(4)
W-C(10)	2.356(5)
W-C(8)	2.361(5)
W-C(7)	2.362(4)
W-C(9)	2.363(5)
W-C(6)	2.377(5)
C(6)-C(7)	1.373(8)
C(6)-C(10)	1.398(9)
C(7)-C(8)	1.377(7)
C(8)-C(9)	1.392(9)
C(9)-C(10)	1.409(10)
C(1)-C(2)	1.422(5)
C(1)-C(4)	1.557(5)
C(2)-C(3)	1.394(5)
C(2)-C(12)	1.484(5)
C(12)-C(13)	1.386(5)
C(12)-C(17)	1.390(6)
C(13)-C(14)	1.378(6)
C(14)-C(15)	1.366(7)
C(15)-C(16)	1.374(7)
C(16)-C(17)	1.379(7)
C(3)-N(2)	1.316(5)
C(3)-C(4)	1.524(5)
N(2)-C(20)	1.461(5)
N(2)-C(18)	1.462(5)
C(18)-C(19)	1.505(7)
C(20)-C(21)	1.513(8)
C(4)-C(11)	1.516(6)
N(1)-O(1)	1.215(4)
O(2)-C(5)	1.138(6)
N(1)-W-C(5)	91.9(2)

N(1)-W-C(1)	96.3(2)
C(5)-W-C(1)	87.8(2)
N(1)-W-C(10)	132.9(2)
C(5)-W-C(10)	92.6(2)
C(1)-W-C(10)	130.7(2)
N(1)-W-C(8)	104.6(2)
C(5)-W-C(8)	149.0(2)
C(1)-W-C(8)	115.6(2)
C(10)-W-C(8)	56.9(2)
N(1)-W-C(7)	132.8(2)
C(5)-W-C(7)	135.2(2)
C(1)-W-C(7)	90.5(1)
C(10)-W-C(7)	56.4(2)
C(8)-W-C(7)	33.9(2)
N(1)-W-C(9)	104.2(2)
C(5)-W-C(9)	116.6(2)
C(1)-W-C(9)	147.0(2)
C(10)-W-C(9)	34.7(2)
C(8)-W-C(9)	34.3(2)
C(7)-W-C(9)	56.6(2)
N(1)-W-C(6)	160.4(2)
C(5)-W-C(6)	102.4(2)
C(1)-W-C(6)	97.7(2)
C(10)-W-C(6)	34.3(2)
C(8)-W-C(6)	56.7(2)
C(7)-W-C(6)	33.7(2)
C(9)-W-C(6)	57.3(2)
C(7)-C(6)-C(10)	107.1(5)
C(7)-C(6)-W	72.6(3)
C(10)-C(6)-W	72.0(3)
C(6)-C(7)-C(8)	109.7(5)
C(6)-C(7)-W	73.7(3)
C(8)-C(7)-W	73.0(3)
C(7)-C(8)-C(9)	108.1(5)
C(7)-C(8)-W	73.1(3)
C(9)-C(8)-W	73.0(3)

C(8)-C(9)-C(10)	106.8(5)
C(8)-C(9)-W	72.8(3)
C(10)-C(9)-W	72.3(3)
C(6)-C(10)-C(9)	108.2(5)
C(6)-C(10)-W	73.6(3)
C(9)-C(10)-W	72.9(3)
C(2)-C(1)-C(4)	90.2(3)
C(2)-C(1)-W	139.5(3)
C(4)-C(1)-W	130.3(2)
C(3)-C(2)-C(1)	93.5(3)
C(3)-C(2)-C(12)	135.3(3)
C(1)-C(2)-C(12)	131.1(4)
C(13)-C(12)-C(17)	118.2(4)
C(13)-C(12)-C(2)	121.0(4)
C(17)-C(12)-C(2)	120.7(4)
C(14)-C(13)-C(12)	120.8(4)
C(15)-C(14)-C(13)	120.4(4)
C(14)-C(15)-C(16)	119.6(4)
C(15)-C(16)-C(17)	120.5(5)
C(16)-C(17)-C(12)	120.4(4)
N(2)-C(3)-C(2)	136.4(3)
N(2)-C(3)-C(4)	130.9(3)
C(2)-C(3)-C(4)	92.7(3)
C(3)-N(2)-C(20)	119.4(3)
C(3)-N(2)-C(18)	122.5(3)
C(20)-N(2)-C(18)	117.8(3)
N(2)-C(18)-C(19)	112.6(4)
N(2)-C(20)-C(21)	112.2(4)
C(11)-C(4)-C(3)	118.2(4)
C(11)-C(4)-C(1)	115.5(3)
C(3)-C(4)-C(1)	83.5(3)
O(1)-N(1)-W	175.0(3)
O(2)-C(5)-W	178.0(5)

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

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
W	42(1)	35(1)	40(1)	3(1)	13(1)	3(1)
C(6)	50(3)	56(3)	77(4)	22(3)	14(3)	7(2)
C(7)	70(3)	44(2)	53(3)	8(2)	24(3)	7(2)
C(8)	56(3)	46(2)	85(4)	14(2)	22(3)	3(2)
C(9)	126(5)	56(3)	78(4)	26(3)	70(4)	28(3)
C(10)	131(6)	56(3)	41(3)	9(2)	-16(4)	1(3)
C(1)	35(2)	38(2)	39(2)	3(2)	11(2)	5(1)
C(2)	37(2)	40(2)	40(2)	0(2)	12(2)	7(2)
C(12)	35(2)	40(2)	43(2)	4(2)	15(2)	7(2)
C(13)	40(2)	45(2)	56(3)	-2(2)	15(2)	2(2)
C(14)	54(3)	35(2)	83(4)	1(2)	33(3)	4(2)
C(15)	50(3)	56(3)	85(4)	15(2)	25(3)	23(2)
C(16)	39(2)	64(3)	99(5)	-7(3)	1(3)	10(2)
C(17)	49(2)	46(2)	68(3)	-6(2)	4(2)	9(2)
C(3)	36(2)	38(2)	40(2)	-1(2)	13(2)	6(1)
N(2)	45(2)	44(2)	42(2)	5(1)	17(2)	8(1)
C(18)	57(3)	44(2)	49(3)	3(2)	16(2)	5(2)
C(19)	75(4)	91(4)	51(3)	-5(3)	8(3)	15(3)
C(20)	54(2)	55(2)	59(3)	4(2)	33(2)	14(2)
C(21)	57(3)	97(4)	99(5)	-9(4)	36(3)	1(3)
C(4)	40(2)	43(2)	44(2)	5(2)	16(2)	11(2)
C(11)	40(2)	79(3)	59(3)	20(2)	12(2)	16(2)
N(1)	50(2)	35(2)	53(2)	5(2)	23(2)	-2(1)
O(1)	45(2)	55(2)	94(3)	12(2)	31(2)	9(1)
O(2)	88(3)	66(2)	126(5)	-19(3)	-2(3)	-20(2)
C(5)	52(3)	48(2)	69(3)	0(2)	11(3)	-2(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **4a**.

	x	y	z	U(eq)
H(1)	811	10818	1013	75
H(7)	2968	11447	2213	65
H(8)	5123	11437	1627	75
H(9)	4333	10787	24	93
H(10)	1643	10386	-350	103
H(13)	2063	11311	3739	56
H(14)	206	12360	3321	66
H(15)	-2058	12009	2279	75
H(16)	-2529	10570	1737	86
H(17)	-692	9505	2155	69
H(18A)	3810	7690	4867	60
H(18B)	2511	7557	5297	60
H(19A)	4710	7813	6483	112
H(19B)	3603	8595	6462	112
H(19C)	4902	8729	6032	112
H(20A)	890	9663	4584	63
H(20B)	1272	9076	5490	63
H(21A)	-1118	8748	4533	122
H(21B)	-115	7900	4627	122
H(21C)	-490	8483	3718	122
H(4)	3725	8097	3195	50
H(11A)	6117	8670	3618	90
H(11B)	5748	8589	4564	90
H(11C)	5646	9518	4068	90

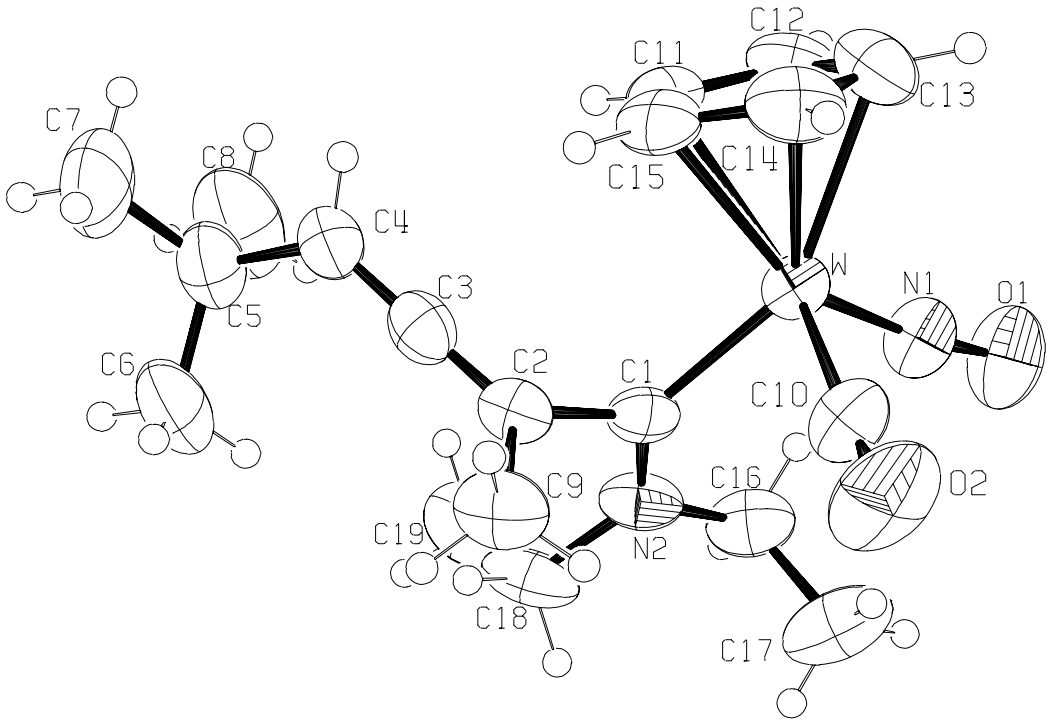

Figure 2. Molecular structure and atom numbering scheme for **7a** with H atoms. Thermal ellipsoids shown at 40%.

Table 6. Crystal Data and Conditions for Crystallographic Data Collection and Structure Refinement for **7a**.

Formula	$C_{19}H_{28}N_2O_2W$
Fw	500.28
Color	red, transparent
cryst syst	monoclinic
space group	$P2_1/c$
cell params	a = 7.838(1) Å, $\alpha = 90°$
	b = 14.031(2) Å, β = 98.59(2)°
	c = 18.567(1) Å, $\gamma = 90°$
V/ Å^3	2019.09
Z	2
d_{calc}/g.cm^{-3}	1.646
μ/cm^{-1}	57.3
diffractometer	Image Plate Diffractometer system (STOE)
radiation	Mo K_α
monochromator	graphite
2θ/deg	$3.27° \leq 2\theta \leq 52.10°$
no. Of reflns meads	14726
no. of indep rflns	3625
R_{int}	0.0283
reflns with $F_o > 4\sigma(F_o)$	3103
temperature/K	293
applied corrections	Lorenz and polarization coefficients
structure determination and refinement	W positional params from direct methods (SHELX-97);[a]
	further atoms from ΔF synthesis (SHELX-97),[b] refinement
	by anisotropic full-matrix least-squares procedure for
	all non-hydrogens; hydrogen position refinement by riding
	model; atomic scattering factors from literature. [c]
refined params	245
wR2	0.066
R1	0.032
R1 [$F_o > 4\sigma(F_o)$]	0.025
max and min in $\Delta\sigma$ (e A^{-3})	0.74, -0.69

[a] Sheldrick, G. M. SHELXS-97, program for the Solution of Crystal Structures; Universität Göttingen, 1997.

[b] Sheldrick, G. M. SHELXL-97, Program for Crystal Structure Reifnement; Universität Göttingen, 1997.

[c] *International Tables for Crystalography*; Wilson , A. J. C., Ed.; Kluwer Academic: Dordrecht , The Netherlands, 1992; Vol. C.

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

	x	y	z	U(eq)
W	3235(1)	4679(1)	3556(1)	42(1)
C(11)	2457(8)	4886(4)	4718(3)	53(1)
C(12)	3163(8)	3993(5)	4700(3)	64(2)
C(13)	2108(9)	3438(5)	4201(4)	68(2)
C(14)	724(8)	4009(5)	3898(3)	66(2)
C(15)	928(7)	4911(4)	4222(3)	54(1)
N(1)	5150(6)	4121(4)	3357(2)	56(1)
O(1)	6430(6)	3655(4)	3272(3)	85(1)
C(10)	2139(8)	4690(5)	2536(3)	61(1)
O(2)	1383(8)	4658(4)	1954(2)	102(2)
C(1)	3903(7)	6129(4)	3445(2)	46(1)
N(2)	5349(6)	6548(3)	3292(2)	54(1)
C(16)	6854(8)	5984(5)	3163(3)	68(2)
C(17)	6778(13)	5703(8)	2374(4)	105(3)
C(18)	5584(9)	7588(5)	3235(3)	71(2)
C(19)	6687(10)	7982(6)	3906(4)	89(2)
C(2)	2459(7)	6823(4)	3504(3)	47(1)
C(9)	1256(8)	7055(5)	2797(3)	65(2)
C(3)	2217(7)	7176(4)	4129(3)	53(1)
C(4)	1845(9)	7502(4)	4738(3)	67(2)
C(5)	2286(10)	8460(4)	5076(4)	71(2)
C(6)	2614(13)	9179(5)	4502(5)	91(2)
C(7)	757(13)	8776(6)	5441(6)	103(3)
C(8)	3893(15)	8382(7)	5648(6)	116(3)

Table 8. Bond lengths [Å] and angles [°] for ipak069j.

W-N(1)	1.780(5)
W-C(10)	1.958(6)
W-C(1)	2.118(5)
W-C(12)	2.342(5)
W-C(11)	2.348(5)
W-C(14)	2.353(6)
W-C(13)	2.360(6)
W-C(15)	2.361(6)
C(11)-C(12)	1.373(9)
C(11)-C(15)	1.399(8)
C(12)-C(13)	1.386(9)
C(13)-C(14)	1.397(9)
C(14)-C(15)	1.401(9)
N(1)-O(1)	1.227(6)
C(10)-O(2)	1.154(7)
C(1)-N(2)	1.345(7)
C(1)-C(2)	1.509(8)
N(2)-C(16)	1.470(8)
N(2)-C(18)	1.476(8)
C(16)-C(17)	1.509(9)
C(18)-C(19)	1.511(9)
C(2)-C(3)	1.301(7)
C(2)-C(9)	1.532(7)
C(3)-C(4)	1.293(8)
C(4)-C(5)	1.503(8)
C(5)-C(6)	1.518(10)
C(5)-C(8)	1.528(11)
C(5)-C(7)	1.525(10)
N(1)-W-C(10)	93.7(2)
N(1)-W-C(1)	100.1(2)
C(10)-W-C(1)	88.7(2)
N(1)-W-C(12)	98.2(2)
C(10)-W-C(12)	144.5(3)

C(1)-W-C(12)	121.5(2)
N(1)-W-C(11)	126.0(2)
C(10)-W-C(11)	138.7(2)
C(1)-W-C(11)	94.15(19)
C(12)-W-C(11)	34.0(2)
N(1)-W-C(14)	130.2(3)
C(10)-W-C(14)	90.0(2)
C(1)-W-C(14)	129.7(2)
C(12)-W-C(14)	57.0(2)
C(11)-W-C(14)	57.2(2)
N(1)-W-C(13)	99.9(2)
C(10)-W-C(13)	110.7(3)
C(1)-W-C(13)	151.0(2)
C(12)-W-C(13)	34.3(2)
C(11)-W-C(13)	57.0(2)
C(14)-W-C(13)	34.5(2)
N(1)-W-C(15)	154.8(2)
C(10)-W-C(15)	104.2(2)
C(1)-W-C(15)	97.9(2)
C(12)-W-C(15)	57.1(2)
C(11)-W-C(15)	34.6(2)
C(14)-W-C(15)	34.6(2)
C(13)-W-C(15)	57.4(2)
C(12)-C(11)-C(15)	108.4(5)
C(12)-C(11)-W	72.8(3)
C(15)-C(11)-W	73.3(3)
C(11)-C(12)-C(13)	109.1(6)
C(11)-C(12)-W	73.2(3)
C(13)-C(12)-W	73.6(3)
C(12)-C(13)-C(14)	107.3(6)
C(12)-C(13)-W	72.2(3)
C(14)-C(13)-W	72.5(3)
C(13)-C(14)-C(15)	108.2(5)
C(13)-C(14)-W	73.0(4)
C(15)-C(14)-W	73.0(3)
C(11)-C(15)-C(14)	107.0(5)

C(11)-C(15)-W	72.2(3)
C(14)-C(15)-W	72.4(3)
O(1)-N(1)-W	172.7(5)
O(2)-C(10)-W	174.5(6)
N(2)-C(1)-C(2)	113.5(4)
N(2)-C(1)-W	132.0(4)
C(2)-C(1)-W	114.5(4)
C(1)-N(2)-C(16)	121.4(5)
C(1)-N(2)-C(18)	124.4(5)
C(16)-N(2)-C(18)	114.1(5)
N(2)-C(16)-C(17)	112.5(6)
N(2)-C(18)-C(19)	111.4(5)
C(3)-C(2)-C(1)	121.4(4)
C(3)-C(2)-C(9)	122.0(5)
C(1)-C(2)-C(9)	116.6(5)
C(4)-C(3)-C(2)	175.1(7)
C(3)-C(4)-C(5)	127.9(6)
C(4)-C(5)-C(6)	110.7(6)
C(4)-C(5)-C(8)	110.0(6)
C(6)-C(5)-C(8)	109.1(8)
C(4)-C(5)-C(7)	107.4(6)
C(6)-C(5)-C(7)	110.2(7)
C(8)-C(5)-C(7)	109.5(8)

Table 9. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for ipak069j. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
W	32(1)	53(1)	39(1)	-1(1)	1(1)	1(1)
C(11)	54(4)	65(3)	42(3)	2(2)	12(2)	-11(3)
C(12)	59(4)	74(4)	56(3)	27(3)	-1(3)	-6(3)
C(13)	66(4)	54(3)	86(4)	16(3)	14(3)	-4(3)
C(14)	45(4)	84(4)	67(3)	8(3)	2(3)	-22(3)
C(15)	34(3)	72(4)	60(3)	13(3)	15(2)	0(2)
N(1)	36(3)	71(3)	59(3)	-9(2)	-1(2)	5(2)
O(1)	50(3)	97(4)	105(3)	-21(3)	8(2)	25(2)
C(10)	52(4)	78(4)	53(3)	-10(3)	6(3)	5(3)
O(2)	100(4)	149(5)	45(2)	-16(3)	-24(2)	26(4)
C(1)	50(3)	56(3)	29(2)	6(2)	-2(2)	-4(2)
N(2)	37(3)	72(3)	52(2)	20(2)	3(2)	-6(2)
C(16)	45(4)	95(5)	63(3)	11(3)	9(3)	-7(3)
C(17)	87(7)	165(9)	67(4)	4(5)	30(4)	1(6)
C(18)	51(4)	81(4)	78(4)	40(3)	3(3)	-16(3)
C(19)	51(5)	94(5)	117(6)	17(4)	-4(4)	-24(4)
C(2)	35(3)	53(3)	53(3)	10(2)	2(2)	-2(2)
C(9)	42(4)	85(4)	64(3)	17(3)	-6(3)	4(3)
C(3)	45(3)	47(3)	65(3)	7(2)	3(2)	5(2)
C(4)	94(5)	47(3)	62(3)	1(3)	19(3)	8(3)
C(5)	82(5)	54(3)	72(4)	-8(3)	-1(3)	8(3)
C(6)	116(7)	48(4)	106(6)	2(4)	3(5)	6(4)
C(7)	125(8)	71(5)	115(6)	-35(5)	27(6)	7(5)
C(8)	133(9)	91(6)	107(6)	-7(6)	-37(6)	13(6)

Table 10. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **7a**.

	x	y	z	U(eq)
H(11)	2917	5389	5011	120(30)
H(12)	4186	3792	4979	80(20)
H(13)	2287	2803	4089	100(30)
H(14)	-177	3821	3542	80(20)
H(15)	189	5429	4126	80(20)
H(16A)	6918	5412	3459	66(18)
H(16B)	7895	6351	3313	75(19)
H(17A)	7780	5335	2315	110(30)
H(17B)	6742	6267	2080	90(30)
H(17C)	5760	5330	2226	90(30)
H(18A)	4465	7898	3173	100(30)
H(18B)	6122	7727	2809	90(20)
H(19A)	6803	8659	3855	130(30)
H(19B)	7807	7691	3960	90(20)
H(19C)	6154	7847	4327	130(40)
H(9A)	1650	6729	2398	230(60)
H(9B)	1259	7730	2712	240(60)
H(9C)	106	6850	2838	210(60)
H(4)	1232	7092	4998	48(14)
H(6A)	2888	9786	4727	110(30)
H(6B)	1601	9238	4144	120(30)
H(6C)	3563	8966	4271	110(30)
H(7A)	994	9392	5658	130(30)
H(7B)	584	8324	5812	120(30)
H(7C)	-265	8810	5085	180(50)
H(8A)	4174	8998	5859	170(40)
H(8B)	4840	8153	5423	170(50)
H(8C)	3675	7946	6022	100(30)

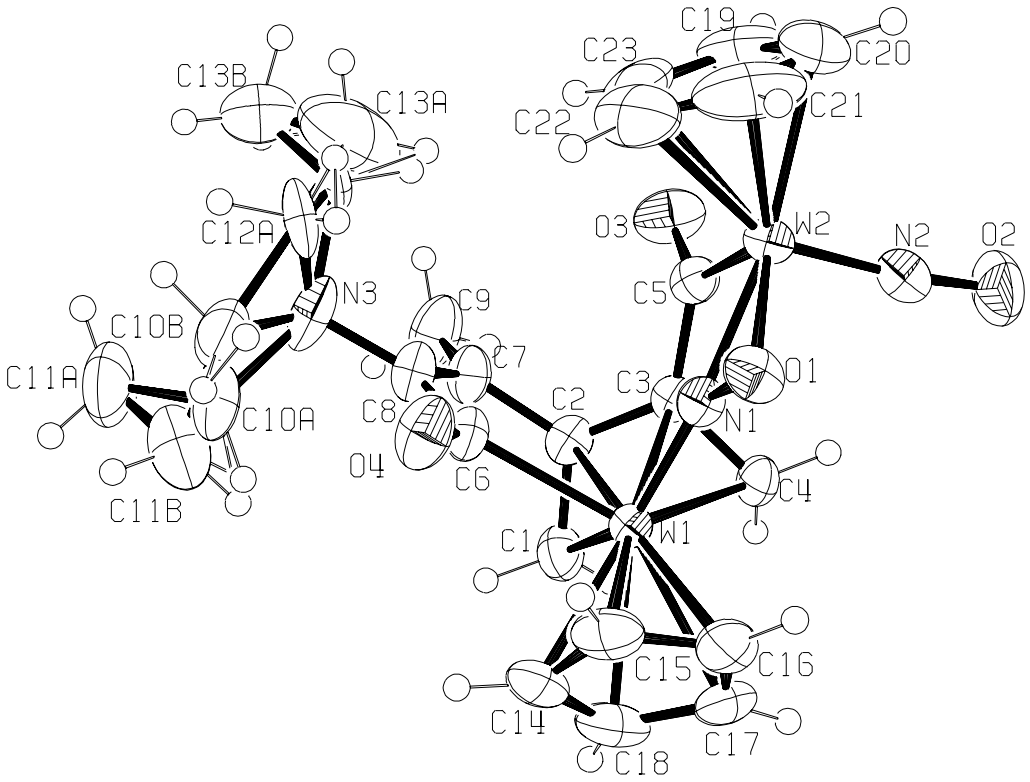

Figure 3. Molecular structure and atom numbering scheme for **9** with H atoms. Thermal ellipsoids shown at 40%.

Table 11. Crystal Data and Conditions for Crystallographic Data Collection and Structure Refinement for **9**.

Formula	$C_{23}H_{27}N_3O_4W_2$
Fw	777.18
Color	red, transparent
cryst syst	monoclinic
space group	$P2_1/n$
cell params	a = 7.665(1) Å, α = 90°
	b = 19.017(2) Å, β = 98.941(7)°
	c = 16.297(1) Å, γ = 90°
V/ Å^3	2346.78
Z	4
d_{calc}/g.cm^{-3}	2.435
μ/cm^{-1}	98.3
diffractometer	Image Plate Diffractometer system (STOE)
radiation	Mo K_α
monochromator	graphite
2θ/deg	$3.27° \leq 2\theta \leq 52.10°$
no. Of reflns meads	17444
no. of indep rflns	4317
R_{int}	0.0540
reflns with $F_\circ > 4\sigma(F_\circ)$	3410
temperature/K	293
applied corrections	Lorenz and polarization coefficients
structure determination and refinement	W positional params from direct methods (SHELX-97);[a]
	further atoms from ΔF synthesis (SHELX-97),[b] refinement
	by anisotropic full-matrix least-squares procedure
	for all non-hydrogens and H(1A), H(1B), H(4A) and
	H(4B); the residue hydrogen atoms position refinement by
	riding model; atomic scattering factors from literature. [c]
refined params	342
wR2	0.052
R1	0.038
R1 [$F_\circ > 4\sigma(F_\circ)$]	0.024
max and min in $\Delta\sigma$ (e A^{-3})	0.79, -0.54

[a] Sheldrick, G. M. SHELXS-97, program for the Solution of Crystal Structures; Universität Göttingen, 1997.

[b] Sheldrick, G. M. SHELXL-97, Program for Crystal Structure Reifnement; Universität Göttingen, 1997.

[c] *International Tables for Crystalography*; Wilson , A. J. C., Ed.; Kluwer Academic: Dordrecht , The Netherlands, 1992; Vol. C.

Table 12. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **9**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
W(1)	2339(1)	1451(1)	702(1)	31(1)
W(2)	3122(1)	1249(1)	-1501(1)	33(1)
O(3)	-398(7)	2055(2)	-1726(3)	57(1)
C(5)	639(8)	1703(3)	-1254(4)	37(1)
C(4)	64(9)	882(3)	-61(4)	37(1)
C(3)	157(7)	1580(3)	-402(3)	31(1)
C(1)	-252(10)	1929(4)	981(4)	45(2)
C(2)	-63(8)	2130(3)	165(4)	36(1)
C(7)	251(9)	2886(3)	-6(4)	41(2)
C(9)	-1368(10)	3314(3)	-308(6)	66(2)
C(8)	1883(9)	3113(3)	212(4)	44(2)
N(3)	2372(9)	3822(3)	111(5)	64(2)
C(6)	3208(9)	2561(3)	558(4)	38(1)
O(4)	4770(6)	2700(2)	729(3)	54(1)
C(10A)	3420(30)	4090(10)	1015(17)	61(6)
C(11A)	2870(30)	4838(9)	1167(13)	85(6)
C(12A)	3250(40)	4022(11)	-446(15)	99(9)
C(13A)	2180(50)	4020(20)	-1227(18)	138(15)
C(10B)	3750(30)	4179(10)	555(13)	68(6)
C(11B)	3220(40)	4230(14)	1415(15)	82(8)
C(12B)	2530(50)	3955(11)	-832(13)	96(11)
C(13B)	1900(40)	4652(12)	-1154(12)	106(8)
N(2)	2046(8)	431(3)	-1706(3)	44(1)
O(2)	1333(8)	-126(2)	-1928(3)	68(2)
N(1)	3455(7)	1275(2)	-245(3)	32(1)
O(1)	4911(6)	982(2)	-455(2)	42(1)
C(14)	3817(10)	1576(4)	2040(4)	51(2)
C(15)	4941(9)	1240(4)	1560(4)	50(2)
C(16)	4211(11)	580(3)	1315(4)	56(2)
C(17)	2631(12)	505(3)	1646(4)	56(2)
C(18)	2402(12)	1114(4)	2088(4)	60(2)

C(19)	2756(12)	1841(5)	-2760(5)	69(2)
C(20)	4104(17)	1373(5)	-2781(5)	82(3)
C(21)	5470(13)	1578(7)	-2187(7)	86(3)
C(22)	5008(16)	2166(6)	-1812(5)	86(3)
C(23)	3347(15)	2329(4)	-2176(6)	75(3)

Table 13. Bond lengths [Å] and angles [°] for **9**.

W(1)-N(1)	1.908(5)
W(1)-C(6)	2.238(5)
W(1)-C(4)	2.255(6)
W(1)-C(3)	2.271(5)
W(1)-C(15)	2.286(6)
W(1)-C(1)	2.293(7)
W(1)-C(14)	2.307(6)
W(1)-C(2)	2.307(6)
W(1)-C(16)	2.313(6)
W(1)-C(18)	2.341(6)
W(1)-C(17)	2.353(6)
W(2)-N(2)	1.768(5)
W(2)-N(1)	2.023(5)
W(2)-O(1)	2.077(4)
W(2)-C(5)	2.183(6)
W(2)-C(19)	2.320(6)
W(2)-C(20)	2.338(8)
W(2)-C(21)	2.348(9)
W(2)-C(23)	2.350(7)
W(2)-C(22)	2.371(8)
O(3)-C(5)	1.217(7)
C(5)-C(3)	1.511(8)
C(4)-C(3)	1.445(7)
C(3)-C(2)	1.423(8)
C(1)-C(2)	1.412(9)
C(2)-C(7)	1.490(8)
C(7)-C(8)	1.319(9)
C(7)-C(9)	1.501(9)
C(8)-N(3)	1.415(8)
C(8)-C(6)	1.508(8)
N(3)-C(12A)	1.27(3)
N(3)-C(10B)	1.36(2)
N(3)-C(12B)	1.58(2)
N(3)-C(10A)	1.64(2)

C(6)-O(4)	1.215(7)
C(10A)-C(11A)	1.52(2)
C(12A)-C(13A)	1.40(3)
C(10B)-C(11B)	1.52(3)
C(12B)-C(13B)	1.48(3)
N(2)-O(2)	1.220(6)
N(1)-O(1)	1.338(6)
C(14)-C(15)	1.403(10)
C(14)-C(18)	1.408(11)
C(15)-C(16)	1.406(10)
C(16)-C(17)	1.407(11)
C(17)-C(18)	1.389(10)
C(19)-C(23)	1.356(12)
C(19)-C(20)	1.368(13)
C(20)-C(21)	1.364(14)
C(21)-C(22)	1.348(15)
C(22)-C(23)	1.354(14)

N(1)-W(1)-C(6)	84.4(2)
N(1)-W(1)-C(4)	82.8(2)
C(6)-W(1)-C(4)	127.7(2)
N(1)-W(1)-C(3)	75.2(2)
C(6)-W(1)-C(3)	90.5(2)
C(4)-W(1)-C(3)	37.2(2)
N(1)-W(1)-C(15)	90.3(2)
C(6)-W(1)-C(15)	89.1(2)
C(4)-W(1)-C(15)	141.2(2)
C(3)-W(1)-C(15)	165.5(2)
N(1)-W(1)-C(1)	137.8(2)
C(6)-W(1)-C(1)	85.9(3)
C(4)-W(1)-C(1)	71.3(3)
C(3)-W(1)-C(1)	63.9(2)
C(15)-W(1)-C(1)	130.5(3)
N(1)-W(1)-C(14)	124.5(2)
C(6)-W(1)-C(14)	83.8(2)
C(4)-W(1)-C(14)	142.6(2)

C(3)-W(1)-C(14)	158.5(2)
C(15)-W(1)-C(14)	35.6(3)
C(1)-W(1)-C(14)	94.9(3)
N(1)-W(1)-C(2)	102.8(2)
C(6)-W(1)-C(2)	70.4(2)
C(4)-W(1)-C(2)	63.6(2)
C(3)-W(1)-C(2)	36.2(2)
C(15)-W(1)-C(2)	154.0(2)
C(1)-W(1)-C(2)	35.8(2)
C(14)-W(1)-C(2)	123.2(2)
N(1)-W(1)-C(16)	84.1(2)
C(6)-W(1)-C(16)	123.1(3)
C(4)-W(1)-C(16)	105.6(2)
C(3)-W(1)-C(16)	138.6(2)
C(15)-W(1)-C(16)	35.6(2)
C(1)-W(1)-C(16)	134.2(3)
C(14)-W(1)-C(16)	59.0(3)
C(2)-W(1)-C(16)	165.8(3)
N(1)-W(1)-C(18)	142.0(2)
C(6)-W(1)-C(18)	113.5(2)
C(4)-W(1)-C(18)	107.4(3)
C(3)-W(1)-C(18)	134.1(3)
C(15)-W(1)-C(18)	58.5(3)
C(1)-W(1)-C(18)	78.8(3)
C(14)-W(1)-C(18)	35.3(3)
C(2)-W(1)-C(18)	114.5(3)
C(16)-W(1)-C(18)	58.0(3)
N(1)-W(1)-C(17)	112.7(2)
C(6)-W(1)-C(17)	142.2(2)
C(4)-W(1)-C(17)	88.9(2)
C(3)-W(1)-C(17)	125.7(2)
C(15)-W(1)-C(17)	58.8(3)
C(1)-W(1)-C(17)	99.9(3)
C(14)-W(1)-C(17)	58.6(2)
C(2)-W(1)-C(17)	131.7(3)
C(16)-W(1)-C(17)	35.1(3)

C(18)-W(1)-C(17)	34.4(3)
N(2)-W(2)-N(1)	101.3(2)
N(2)-W(2)-O(1)	99.8(2)
N(1)-W(2)-O(1)	38.06(17)
N(2)-W(2)-C(5)	89.3(2)
N(1)-W(2)-C(5)	77.3(2)
O(1)-W(2)-C(5)	115.39(19)
N(2)-W(2)-C(19)	105.7(3)
N(1)-W(2)-C(19)	149.5(3)
O(1)-W(2)-C(19)	144.9(2)
C(5)-W(2)-C(19)	89.0(3)
N(2)-W(2)-C(20)	97.2(3)
N(1)-W(2)-C(20)	153.2(3)
O(1)-W(2)-C(20)	119.6(3)
C(5)-W(2)-C(20)	122.4(3)
C(19)-W(2)-C(20)	34.2(3)
N(2)-W(2)-C(21)	120.9(4)
N(1)-W(2)-C(21)	119.3(3)
O(1)-W(2)-C(21)	89.9(3)
C(5)-W(2)-C(21)	137.5(3)
C(19)-W(2)-C(21)	56.3(3)
C(20)-W(2)-C(21)	33.9(4)
N(2)-W(2)-C(23)	138.3(3)
N(1)-W(2)-C(23)	116.4(3)
O(1)-W(2)-C(23)	120.7(3)
C(5)-W(2)-C(23)	82.5(3)
C(19)-W(2)-C(23)	33.8(3)
C(20)-W(2)-C(23)	55.7(3)
C(21)-W(2)-C(23)	55.0(4)
N(2)-W(2)-C(22)	152.7(3)
N(1)-W(2)-C(22)	102.3(3)
O(1)-W(2)-C(22)	90.5(3)
C(5)-W(2)-C(22)	109.1(4)
C(19)-W(2)-C(22)	56.4(3)
C(20)-W(2)-C(22)	56.1(3)
C(21)-W(2)-C(22)	33.2(4)

C(23)-W(2)-C(22)	33.3(4)
O(3)-C(5)-C(3)	115.9(6)
O(3)-C(5)-W(2)	127.2(5)
C(3)-C(5)-W(2)	116.9(4)
C(3)-C(4)-W(1)	72.0(3)
C(2)-C(3)-C(4)	114.1(5)
C(2)-C(3)-C(5)	123.6(5)
C(4)-C(3)-C(5)	122.0(5)
C(2)-C(3)-W(1)	73.3(3)
C(4)-C(3)-W(1)	70.8(3)
C(5)-C(3)-W(1)	119.4(4)
C(2)-C(1)-W(1)	72.7(4)
C(1)-C(2)-C(3)	116.8(5)
C(1)-C(2)-C(7)	118.7(5)
C(3)-C(2)-C(7)	123.3(6)
C(1)-C(2)-W(1)	71.5(4)
C(3)-C(2)-W(1)	70.5(3)
C(7)-C(2)-W(1)	117.9(4)
C(8)-C(7)-C(2)	116.1(5)
C(8)-C(7)-C(9)	127.7(6)
C(2)-C(7)-C(9)	115.8(6)
C(7)-C(8)-N(3)	122.6(6)
C(7)-C(8)-C(6)	115.3(5)
N(3)-C(8)-C(6)	122.1(6)
C(12A)-N(3)-C(8)	123.2(11)
C(10B)-N(3)-C(8)	127.5(10)
C(10B)-N(3)-C(12B)	105.4(15)
C(8)-N(3)-C(12B)	109.1(8)
C(12A)-N(3)-C(10A)	107.9(14)
C(8)-N(3)-C(10A)	107.0(10)
O(4)-C(6)-C(8)	121.1(5)
O(4)-C(6)-W(1)	118.7(4)
C(8)-C(6)-W(1)	120.1(4)
C(11A)-C(10A)-N(3)	109.1(16)
N(3)-C(12A)-C(13A)	110(3)
N(3)-C(10B)-C(11B)	103(2)

C(13B)-C(12B)-N(3)	114.6(18)
O(2)-N(2)-W(2)	173.6(5)
O(1)-N(1)-W(1)	140.9(3)
O(1)-N(1)-W(2)	73.2(3)
W(1)-N(1)-W(2)	145.2(3)
N(1)-O(1)-W(2)	68.8(3)
C(15)-C(14)-C(18)	107.0(6)
C(15)-C(14)-W(1)	71.4(3)
C(18)-C(14)-W(1)	73.7(4)
C(14)-C(15)-C(16)	108.2(7)
C(14)-C(15)-W(1)	73.0(4)
C(16)-C(15)-W(1)	73.2(4)
C(17)-C(16)-C(15)	108.0(6)
C(17)-C(16)-W(1)	74.0(4)
C(15)-C(16)-W(1)	71.2(3)
C(18)-C(17)-C(16)	107.5(7)
C(18)-C(17)-W(1)	72.3(4)
C(16)-C(17)-W(1)	70.9(3)
C(17)-C(18)-C(11)	109.3(8)
C(17)-C(18)-W(1)	73.3(4)
C(14)-C(18)-W(1)	71.1(4)
C(23)-C(19)-C(20)	107.0(8)
C(23)-C(19)-W(2)	74.3(4)
C(20)-C(19)-W(2)	73.6(4)
C(21)-C(20)-C(19)	107.1(8)
C(21)-C(20)-W(2)	73.4(5)
C(19)-C(20)-W(2)	72.2(5)
C(22)-C(21)-C(20)	109.3(9)
C(22)-C(21)-W(2)	74.3(6)
C(20)-C(21)-W(2)	72.6(5)
C(21)-C(22)-C(23)	106.7(8)
C(21)-C(22)-W(2)	72.4(5)
C(23)-C(22)-W(2)	72.5(5)
C(22)-C(23)-C(19)	109.8(9)
C(22)-C(23)-W(2)	74.2(5)
C(19)-C(23)-W(2)	71.9(4)

Table 14. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **9**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
W(1)	35(1)	28(1)	29(1)	0(1)	5(1)	1(1)
W(2)	31(1)	39(1)	29(1)	1(1)	5(1)	0(1)
O(3)	40(3)	72(3)	58(3)	21(2)	5(2)	16(2)
C(5)	35(4)	37(3)	38(3)	3(2)	2(3)	-3(2)
C(4)	36(4)	31(3)	42(3)	-2(2)	5(3)	-7(2)
C(3)	25(3)	33(3)	35(3)	0(2)	2(2)	-1(2)
C(1)	48(5)	40(3)	49(4)	-8(3)	16(3)	-3(3)
C(2)	29(4)	36(3)	44(3)	2(2)	8(3)	1(2)
C(7)	37(4)	32(3)	55(4)	-5(3)	6(3)	3(2)
C(9)	54(5)	39(3)	101(6)	3(4)	-3(4)	9(3)
C(8)	44(4)	29(3)	57(4)	-3(3)	7(3)	-2(2)
N(3)	64(5)	29(3)	99(5)	6(3)	8(4)	-12(3)
C(6)	42(4)	28(3)	44(3)	-2(2)	6(3)	-6(2)
O(4)	35(3)	45(2)	79(3)	2(2)	-1(3)	-6(2)
C(10A)	67(14)	30(9)	90(20)	-13(12)	14(16)	0(8)
C(11A)	109(17)	45(9)	104(15)	-26(9)	24(12)	-5(9)
C(12A)	170(30)	51(11)	71(15)	-39(11)	7(16)	-41(13)
C(13A)	190(30)	160(30)	70(20)	-10(20)	40(20)	60(30)
C(10B)	85(16)	41(9)	72(14)	6(9)	-6(12)	-16(8)
C(11B)	76(17)	80(18)	88(18)	-30(12)	13(13)	-9(12)
C(12B)	210(30)	47(10)	42(14)	-29(11)	45(17)	-75(16)
C(13B)	170(30)	89(14)	59(12)	17(11)	13(13)	-14(15)
N(2)	45(4)	50(3)	37(3)	-5(2)	8(2)	2(2)
O(2)	76(4)	52(3)	75(4)	-23(3)	11(3)	-20(3)
N(1)	29(3)	32(2)	36(2)	1(2)	3(2)	3(2)
O(1)	35(3)	52(2)	40(2)	-2(2)	9(2)	13(2)
C(14)	62(5)	61(4)	26(3)	2(3)	-6(3)	-2(3)
C(15)	38(4)	68(4)	39(3)	14(3)	-7(3)	-1(3)
C(16)	75(6)	47(4)	41(3)	8(3)	-3(4)	30(3)
C(17)	80(6)	46(4)	39(4)	18(3)	0(4)	-3(3)
C(18)	71(6)	81(5)	31(3)	12(3)	14(3)	6(4)

C(19)	55(6)	107(7)	42(4)	37(4)	-2(4)	-10(5)
C(20)	126(10)	80(6)	53(5)	-6(4)	52(6)	-19(6)
C(21)	43(6)	142(9)	83(7)	54(7)	35(5)	18(6)
C(22)	85(8)	113(8)	57(5)	5(5)	6(5)	-62(6)
C(23)	102(8)	53(4)	81(6)	25(4)	44(6)	-4(5)

Table 15. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **9**.

	x	y	z	U(eq)
H(4A)	-1050(100)	800(30)	250(40)	52(19)
H(4B)	220(100)	510(30)	-440(40)	52(19)
H(1A)	-30(70)	2310(30)	1390(30)	26(14)
H(1B)	-940(90)	1580(30)	1020(40)	33(16)
H(9A)	-2374	3008	-422	99
H(9B)	-1210	3558	-806	99
H(9C)	-1555	3648	111	99
H(10a-A)	3132	3786	1453	73
H(10a-B)	4683	4071	1018	73
H(11a-A)	3486	5000	1691	128
H(11a-B)	1617	4852	1177	128
H(11a-C)	3144	5136	730	128
H(12a-A)	4249	3708	-456	119
H(12a-B)	3710	4491	-318	119
H(13a-A)	2870	4166	-1642	207
H(13a-B)	1219	4343	-1220	207
H(13a-C)	1730	3557	-1351	207
H(10b-A)	3876	4642	322	81
H(10b-B)	4847	3922	571	81
H(11b-A)	4129	4474	1780	122
H(11b-B)	3082	3766	1627	122
H(11b-C)	2131	4483	1381	122
H(12b-A)	3763	3904	-900	115
H(12b-B)	1869	3594	-1166	115
H(13b-A)	2012	4686	-1731	158
H(13b-B)	2593	5014	-849	158
H(13b-C)	683	4708	-1091	158
H(14)	3977	2020	2280	61
H(15)	5988	1422	1428	60
H(16)	4688	251	990	67
H(17)	1879	119	1580	67

H(18)	1462	1202	2372	73
H(19)	1646	1828	-3085	83
H(20)	4092	988	-3133	98
H(21)	6549	1348	-2062	104
H(22)	5696	2412	-1387	103
H(23)	2707	2718	-2044	91