CHEMISTRY OF MATERIALS
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STRUCTURE DETERMINATION SUMMARY

Crystal Data

Empirical Formula	$(N_1 H_4) Na_1 Mo_3 O_{10}$
Color; Habit	WHITE NEDDLE-LIKE PRISM
Crystal size (mm)	0.05 X 0.05 X 0.20
Crystal System	Orthorhombic
Space Group	Pmma
Unit Cell Dimensions	a = 8.407(2) Å
	b = 7.603(2) Å
	c = 14.350(3) Å
Volume	917.2(5) $Å^3$
Z	4
Formula weight	242.4
Density(calc.)	3.511 Mg/m^3
Absorption Coefficient	4.126 mm^{-1}
F(000)	896

Data Collection

Diffractometer Used	Siemens R3m/V
Radiation	MoKα (λ = 0.71073 Å)
Temperature (K)	298
Monochromator	Highly oriented graphite crystal
2θ Range	3.0 to 50.0°
Scan Type	ω
Scan Speed	Variable; 2.49 to 26.04°/min. in ω
Scan Range (ω)	1.20°
Background Measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 25.0% of total scan time
Standard Reflections	3 measured every 100 reflections
Index Ranges	$0 \le h \le 10$, $0 \le k \le 9$ $-17 \le l \le 0$
Reflections Collected	868
Independent Reflections	868
Observed Reflections	701 ($F > 5.0\sigma(F)$)
Absorption Correction	Semi-empirical
Min./Max. Transmission	0.6506 / 0.7411

I-818-3

Solution and Refinement

System Used	Siemens SHELXTL PLUS (PC Version)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Quantity Minimized	$\sum w(F_o - F_c)^2$
Absolute Structure	N/A
Extinction Correction	χ = 0.00035(4), where $F^* = F\,[\,1 + 0.002\chi F^2/\sin(2\theta)\,]^{-1/4}$
Hydrogen Atoms	Riding model, fixed isotropic U
Weighting Scheme	$w^{-1} = \sigma^2(F) + 0.0002F^2$
Number of Parameters Refined	80
Final R Indices (obs. data)	R = 2.35 %, wR = 3.07 %
R Indices (all data)	R = 3.48 %, wR = 6.53 %
Goodness-of-Fit	1.33
Largest and Mean Δ/σ	0.019, 0.001
Data-to-Parameter Ratio	8.8:1
Largest Difference Peak	0.72 $e\text{Å}^{-3}$
Largest Difference Hole	-0.76 $e\text{Å}^{-3}$

I-818-4

Table 1. Atomic coordinates ($x10^4$) and equivalent isotropic displacement coefficients ($Å^2 x10^3$)

	x	y	z	U(eq)
Mo(1)	3012(1)	2500	4248(1)	10(1)
Mo(2)	1145(1)	-16(1)	1069(1)	9(1)
Na(1)	-271(4)	2500	3089(3)	20(1)
O(1)	-1120(7)	2500	-717(4)	11(1)
O(2)	-3919(7)	2500	358(4)	16(2)
O(3)	-2248(7)	2500	1942(4)	16(2)
O(4)	616(7)	2500	938(4)	14(2)
O(5)	1320(4)	-121(5)	-511(3)	12(1)
O(6)	3144(5)	343(6)	1134(3)	19(1)
O(7)	561(5)	-311(5)	2189(3)	19(1)
N(1)	3631(10)	2500	-1041(6)	27(2)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2. Bond lengths (Å)

Mo(1)-O(2A)	1.719 (6)	Mo(1)-O(3A)	1.721 (6)
Mo(1)-O(5A)	1.925 (4)	Mo(1)-O(5B)	1.925 (4)
Mo(2)-O(4)	1.973 (2)	Mo(2)-O(6)	1.705 (5)
Mo(2)-O(7)	1.696 (4)	Mo(2)-O(1A)	1.955 (2)
Na(1)-O(3)	2.339 (7)	Na(1)-O(2A)	2.502 (7)
Na(1)-O(6A)	2.389 (5)	Na(1)-O(6B)	2.389 (5)
O(1)-Mo(2A)	1.955 (2)	O(1)-Mo(2B)	1.955 (2)
O(2)-Mo(1B)	1.719 (6)	O(2)-Na(1A)	2.502 (7)
O(3)-Mo(1B)	1.721 (6)	O(4)-Mo(2C)	1.973 (2)
O(5)-Mo(1A)	1.925 (4)	O(6)-Na(1B)	2.389 (5)

Table 3. Bond angles (°)

O(2A)-Mo(1)-O(3A)	101.9(3)	O(2A)-Mo(1)-O(5A)	102.5(1)
O(3A)-Mo(1)-O(5A)	102.4(1)	O(2A)-Mo(1)-O(5B)	102.5(1)
O(3A)-Mo(1)-O(5B)	102.4(1)	O(5A)-Mo(1)-O(5B)	139.9(2)
O(4)-Mo(2)-O(6)	94.1(2)	O(4)-Mo(2)-O(7)	98.8(2)
O(6)-Mo(2)-O(7)	104.7(2)	O(4)-Mo(2)-O(1A)	155.4(2)
O(6)-Mo(2)-O(1A)	100.3(2)	O(7)-Mo(2)-O(1A)	96.5(2)
O(3)-Na(1)-O(2A)	161.7(3)	O(3)-Na(1)-O(6A)	86.1(2)
O(2A)-Na(1)-O(6A)	80.6(2)	O(3)-Na(1)-O(6B)	86.1(2)
O(2A)-Na(1)-O(6B)	80.6(2)	O(6A)-Na(1)-O(6B)	86.7(2)
Mo(2A)-O(1)-Mo(2B)	150.0(3)	Mo(1B)-O(2)-Na(1A)	97.8(3)
Na(1)-O(3)-Mo(1B)	127.4(3)	Mo(2)-O(4)-Mo(2C)	151.7(3)
Mo(2)-O(6)-Na(1B)	133.3(2)		

I-818-5

Table 4. Anisotropic displacement coefficients ($Å^2 x 10^3$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Mo(1)	9(1)	10(1)	11(1)	0	-1(1)	0
Mo(2)	11(1)	7(1)	9(1)	1(1)	-2(1)	-1(1)
Na(1)	14(2)	28(2)	19(2)	0	-1(2)	0
O(1)	19(3)	6(2)	6(2)	0	-1(2)	0
O(2)	16(3)	20(3)	12(3)	0	0(2)	0
O(3)	12(3)	24(3)	13(3)	0	1(2)	0
O(4)	11(3)	9(3)	23(3)	0	-3(2)	0
O(5)	12(2)	8(2)	16(2)	-2(2)	-1(2)	-2(2)
O(6)	16(2)	17(2)	23(2)	0(2)	-4(2)	-5(2)
O(7)	27(2)	17(2)	13(2)	3(2)	2(2)	-2(2)
N(1)	24(4)	35(4)	24(4)	0	-3(3)	0

The anisotropic displacement exponent takes the form:

$-2\pi^2 (h^2 a^{*2} U_{11} + \ldots + 2hka^*b^* U_{12})$

CM9502333 MO-1

I-818-6

6

STRUCTURE DETERMINATION SUMMARY

Crystal Data

Empirical Formula	$C_{12}H_{36}Mo_8N_4O_{26}$
Color; Habit	DARK GRAY PRISM
Crystal size (mm)	0.30 X 0.30 X 0.40
Crystal System	Triclinic
Space Group	$P\bar{1}$
Unit Cell Dimensions	$\underline{a}$ = 8.267(2) Å
	$\underline{b}$ = 8.986(2) Å
	$\underline{c}$ = 12.714(3) Å
	α = 87.58(3)°
	β = 76.48(3)°
	γ = 67.97(3)°
Volume	850.2(3) $Å^3$
Z	1
Formula weight	1420.0
Density(calc.)	2.773 Mg/m^3
Absorption Coefficient	2.950 mm^{-1}
F(000)	680

Data Collection

Diffractometer Used	Siemens R3m/V
Radiation	MoKα (λ = 0.71073 Å)
Temperature (K)	298
Monochromator	Highly oriented graphite crystal
2θ Range	3.5 to 48.0°
Scan Type	ω
Scan Speed	Variable; 2.49 to 26.04°/min. in ω
Scan Range (ω)	1.20°
Background Measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 25.0% of total scan time
Standard Reflections	3 measured every 97 reflections
Index Ranges	$0 \le h \le 10$, $-10 \le k \le 11$ $-16 \le l \le 16$
Reflections Collected	4206
Independent Reflections	3937 (R_{int} = 3.10%)
Observed Reflections	3660 ($F > 6.0\sigma(F)$)
Absorption Correction	Semi-empirical
Min./Max. Transmission	0.5617 / 0.7653

Solution and Refinement

System Used	Siemens SHELXTL PLUS (PC Version)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Quantity Minimized	$\sum w(F_o - F_c)^2$
Absolute Structure	N/A
Extinction Correction	χ = 0.0036(2), where $F^* = F\ [\ 1 + 0.002\chi F^2/\sin(2\theta)\ ]^{-1/4}$
Hydrogen Atoms	Riding model, fixed isotropic U
Weighting Scheme	$w^{-1} = \sigma^2(F) + 0.0005F^2$
Number of Parameters Refined	227
Final R Indices (obs. data)	R = 2.82 %, wR = 6.36 %
R Indices (all data)	R = 2.99 %, wR = 6.44 %
Goodness-of-Fit	2.38
Largest and Mean Δ/σ	0.001, 0.000
Data-to-Parameter Ratio	16.1:1
Largest Difference Peak	1.51 $e\text{Å}^{-3}$
Largest Difference Hole	-1.52 $e\text{Å}^{-3}$

I-818-9

Table 1. Atomic coordinates ($x10^4$) and equivalent isotropic displacement coefficients ($Å^2 x10^3$)

	x	y	z	U(eq)
Mo(1)	9227(1)	2640(1)	4387(1)	13(1)
Mo(2)	4661(1)	3183(1)	4925(1)	13(1)
Mo(3)	7016(1)	1240(1)	6586(1)	14(1)
Mo(4)	6924(1)	4695(1)	2714(1)	14(1)
O(11)	10529(4)	3728(4)	4041(3)	23(1)
O(12)	8893(4)	2557(3)	5810(2)	17(1)
O(13)	8475(3)	2642(3)	2942(2)	17(1)
O(14)	10773(4)	540(3)	3961(2)	22(1)
O(21)	6917(3)	1575(3)	4904(2)	14(1)
O(22)	3227(4)	2223(3)	5301(3)	24(1)
O(23)	5124(3)	3409(3)	6494(2)	16(1)
O(24)	4800(4)	3522(3)	3540(2)	18(1)
O(31)	7304(4)	1742(4)	7804(3)	24(1)
O(32)	5633(4)	208(4)	6908(3)	28(1)
O(41)	6311(4)	4496(4)	1549(2)	26(1)
O(42)	6795(3)	4583(3)	4516(2)	14(1)
O(43)	8423(4)	5622(3)	2361(3)	24(1)
C(1)	2635(7)	4428(6)	11105(4)	34(2)
C(2)	2381(6)	2872(5)	11134(3)	29(2)
C(3)	2661(7)	2149(6)	10005(4)	36(2)
C(4)	2106(6)	729(6)	10042(3)	33(2)
C(5)	2499(5)	-100(5)	8929(3)	26(1)
C(6)	1322(6)	985(5)	8232(4)	28(2)
N(1)	2264(5)	5130(4)	12221(3)	29(1)
N(6)	1934(5)	319(4)	7092(3)	30(1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2. Bond lengths (Å)

Mo(1)-O(11)	1.685 (4)	Mo(1)-O(12)	1.767 (3)
Mo(1)-O(13)	2.073 (3)	Mo(1)-O(14)	1.855 (2)
Mo(1)-O(21)	2.393 (3)	Mo(1)-O(42)	2.087 (2)
Mo(2)-Mo(3)	3.219 (1)	Mo(2)-O(21)	1.878 (2)
Mo(2)-O(22)	1.687 (4)	Mo(2)-O(23)	2.148 (3)
Mo(2)-O(24)	1.758 (3)	Mo(2)-O(42)	2.478 (3)
Mo(2)-O(42A)	1.973 (2)	Mo(3)-O(12)	2.312 (3)
Mo(3)-O(21)	2.164 (3)	Mo(3)-O(23)	2.010 (2)
Mo(3)-O(31)	1.724 (4)	Mo(3)-O(32)	1.697 (4)
Mo(3)-O(14A)	1.921 (2)	Mo(4)-O(13)	1.867 (2)
Mo(4)-O(24)	2.394 (3)	Mo(4)-O(41)	1.711 (3)
Mo(4)-O(42)	2.268 (3)	Mo(4)-O(43)	1.706 (4)
Mo(4)-O(23A)	1.992 (2)	O(14)-Mo(3A)	1.921 (2)
O(23)-Mo(4A)	1.992 (2)	O(42)-Mo(2A)	1.973 (2)
C(1)-C(2)	1.487 (8)	C(1)-N(1)	1.494 (6)
C(2)-C(3)	1.532 (7)	C(3)-C(4)	1.504 (9)
C(4)-C(5)	1.535 (6)	C(5)-C(6)	1.517 (6)
C(6)-N(6)	1.489 (6)		

I-818-10

11

Table 3. Bond angles (°)

O(11)-Mo(1)-O(12)	104.4(2)	O(11)-Mo(1)-O(13)	99.2(2)
O(12)-Mo(1)-O(13)	153.3(1)	O(11)-Mo(1)-O(14)	104.4(1)
O(12)-Mo(1)-O(14)	100.6(1)	O(13)-Mo(1)-O(14)	85.3(1)
O(11)-Mo(1)-O(21)	169.2(1)	O(12)-Mo(1)-O(21)	76.1(1)
O(13)-Mo(1)-O(21)	78.4(1)	O(14)-Mo(1)-O(21)	86.0(1)
O(11)-Mo(1)-O(42)	95.5(1)	O(12)-Mo(1)-O(42)	92.0(1)
O(13)-Mo(1)-O(42)	73.2(1)	O(14)-Mo(1)-O(42)	152.8(1)
O(21)-Mo(1)-O(42)	73.6(1)	Mo(3)-Mo(2)-O(21)	40.4(1)
Mo(3)-Mo(2)-O(22)	91.3(1)	O(21)-Mo(2)-O(22)	103.7(1)
Mo(3)-Mo(2)-O(23)	37.7(1)	O(21)-Mo(2)-O(23)	75.0(1)
O(22)-Mo(2)-O(23)	97.4(1)	Mo(3)-Mo(2)-O(24)	142.1(1)
O(21)-Mo(2)-O(24)	102.0(1)	O(22)-Mo(2)-O(24)	106.5(2)
O(23)-Mo(2)-O(24)	155.9(1)	Mo(3)-Mo(2)-O(42)	85.1(1)
O(21)-Mo(2)-O(42)	75.0(1)	O(22)-Mo(2)-O(42)	175.7(1)
O(23)-Mo(2)-O(42)	78.2(1)	O(24)-Mo(2)-O(42)	77.9(1)
Mo(3)-Mo(2)-O(42A)	110.6(1)	O(21)-Mo(2)-O(42A)	140.0(1)
O(22)-Mo(2)-O(42A)	103.6(1)	O(23)-Mo(2)-O(42A)	73.0(1)
O(24)-Mo(2)-O(42A)	97.5(1)	O(42)-Mo(2)-O(42A)	75.5(1)
Mo(2)-Mo(3)-O(12)	83.9(1)	Mo(2)-Mo(3)-O(21)	34.2(1)
O(12)-Mo(3)-O(21)	71.3(1)	Mo(2)-Mo(3)-O(23)	40.9(1)
O(12)-Mo(3)-O(23)	81.8(1)	O(21)-Mo(3)-O(23)	72.1(1)
Mo(2)-Mo(3)-O(31)	134.2(1)	O(12)-Mo(3)-O(31)	85.6(1)
O(21)-Mo(3)-O(31)	154.1(1)	O(23)-Mo(3)-O(31)	93.5(1)
Mo(2)-Mo(3)-O(32)	88.3(1)	O(12)-Mo(3)-O(32)	169.0(1)
O(21)-Mo(3)-O(32)	97.9(2)	O(23)-Mo(3)-O(32)	97.4(1)
O(31)-Mo(3)-O(32)	105.4(2)	Mo(2)-Mo(3)-O(14A)	119.1(1)
O(12)-Mo(3)-O(14A)	78.7(1)	O(21)-Mo(3)-O(14A)	85.2(1)
O(23)-Mo(3)-O(14A)	153.9(1)	O(31)-Mo(3)-O(14A)	102.1(1)
O(32)-Mo(3)-O(14A)	98.5(1)	O(13)-Mo(4)-O(24)	79.6(1)
O(13)-Mo(4)-O(41)	105.4(1)	O(24)-Mo(4)-O(41)	84.7(1)
O(13)-Mo(4)-O(42)	73.0(1)	O(24)-Mo(4)-O(42)	71.3(1)
O(41)-Mo(4)-O(42)	155.9(1)	O(13)-Mo(4)-O(43)	99.8(1)
O(24)-Mo(4)-O(43)	169.6(1)	O(41)-Mo(4)-O(43)	105.4(2)
O(42)-Mo(4)-O(43)	98.5(1)	O(13)-Mo(4)-O(23A)	141.3(1)
O(24)-Mo(4)-O(23A)	78.1(1)	O(41)-Mo(4)-O(23A)	103.5(1)
O(42)-Mo(4)-O(23A)	70.1(1)	O(43)-Mo(4)-O(23A)	96.7(1)
Mo(1)-O(12)-Mo(3)	113.9(2)	Mo(1)-O(13)-Mo(4)	110.5(1)
Mo(1)-O(14)-Mo(3A)	158.9(2)	Mo(1)-O(21)-Mo(2)	110.4(1)
Mo(1)-O(21)-Mo(3)	97.6(1)	Mo(2)-O(21)-Mo(3)	105.4(1)
Mo(2)-O(23)-Mo(3)	101.4(1)	Mo(2)-O(23)-Mo(4A)	107.3(1)
Mo(3)-O(23)-Mo(4A)	147.2(1)	Mo(2)-O(24)-Mo(4)	114.9(2)
Mo(1)-O(42)-Mo(2)	100.6(1)	Mo(1)-O(42)-Mo(4)	96.0(1)
Mo(2)-O(42)-Mo(4)	95.5(1)	Mo(1)-O(42)-Mo(2A)	146.3(2)
Mo(2)-O(42)-Mo(2A)	104.5(1)	Mo(4)-O(42)-Mo(2A)	103.5(1)
C(2)-C(1)-N(1)	111.3(4)	C(1)-C(2)-C(3)	113.1(4)
C(2)-C(3)-C(4)	112.7(4)	C(3)-C(4)-C(5)	114.0(4)
C(4)-C(5)-C(6)	110.8(3)	C(5)-C(6)-N(6)	110.7(3)

I-818-11

Table 4. Anisotropic displacement coefficients ($Å^2 x 10^3$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Mo(1)	9(1)	12(1)	16(1)	-1(1)	-3(1)	3(1)
Mo(2)	10(1)	12(1)	14(1)	-2(1)	-3(1)	2(1)
Mo(3)	11(1)	13(1)	15(1)	-1(1)	-2(1)	4(1)
Mo(4)	11(1)	13(1)	13(1)	-1(1)	-2(1)	3(1)
O(11)	19(1)	24(2)	27(2)	-11(1)	-2(1)	6(1)
O(12)	17(1)	18(1)	16(1)	-6(1)	-4(1)	3(1)
O(13)	15(1)	14(1)	17(1)	-3(1)	-2(1)	3(1)
O(14)	15(1)	14(1)	29(2)	3(1)	-3(1)	1(1)
O(21)	13(1)	13(1)	14(1)	-2(1)	-3(1)	2(1)
O(22)	20(1)	23(2)	33(2)	-13(1)	-7(1)	8(1)
O(23)	14(1)	13(1)	15(1)	3(1)	-5(1)	1(1)
O(24)	17(1)	18(1)	17(1)	-5(1)	-4(1)	2(1)
O(31)	23(1)	25(2)	19(1)	-4(1)	-6(1)	6(1)
O(32)	19(1)	24(2)	39(2)	-10(1)	-1(1)	6(1)
O(41)	27(1)	30(2)	18(1)	-7(1)	-7(1)	5(1)
O(42)	10(1)	13(1)	16(1)	-2(1)	-2(1)	-1(1)
O(43)	17(1)	17(1)	34(2)	-5(1)	-4(1)	6(1)
C(1)	40(2)	47(3)	26(2)	-25(2)	-13(2)	10(2)
C(2)	26(2)	32(2)	25(2)	-8(2)	-6(2)	2(2)
C(3)	44(3)	49(3)	21(2)	-27(2)	-5(2)	0(2)
C(4)	40(2)	41(3)	21(2)	-17(2)	-8(2)	2(2)
C(5)	31(2)	22(2)	20(2)	-5(2)	-2(2)	2(1)
C(6)	32(2)	24(2)	27(2)	-8(2)	-10(2)	-2(2)
N(1)	26(2)	32(2)	29(2)	-10(2)	-9(2)	4(2)
N(6)	38(2)	27(2)	28(2)	-15(2)	-10(2)	2(2)

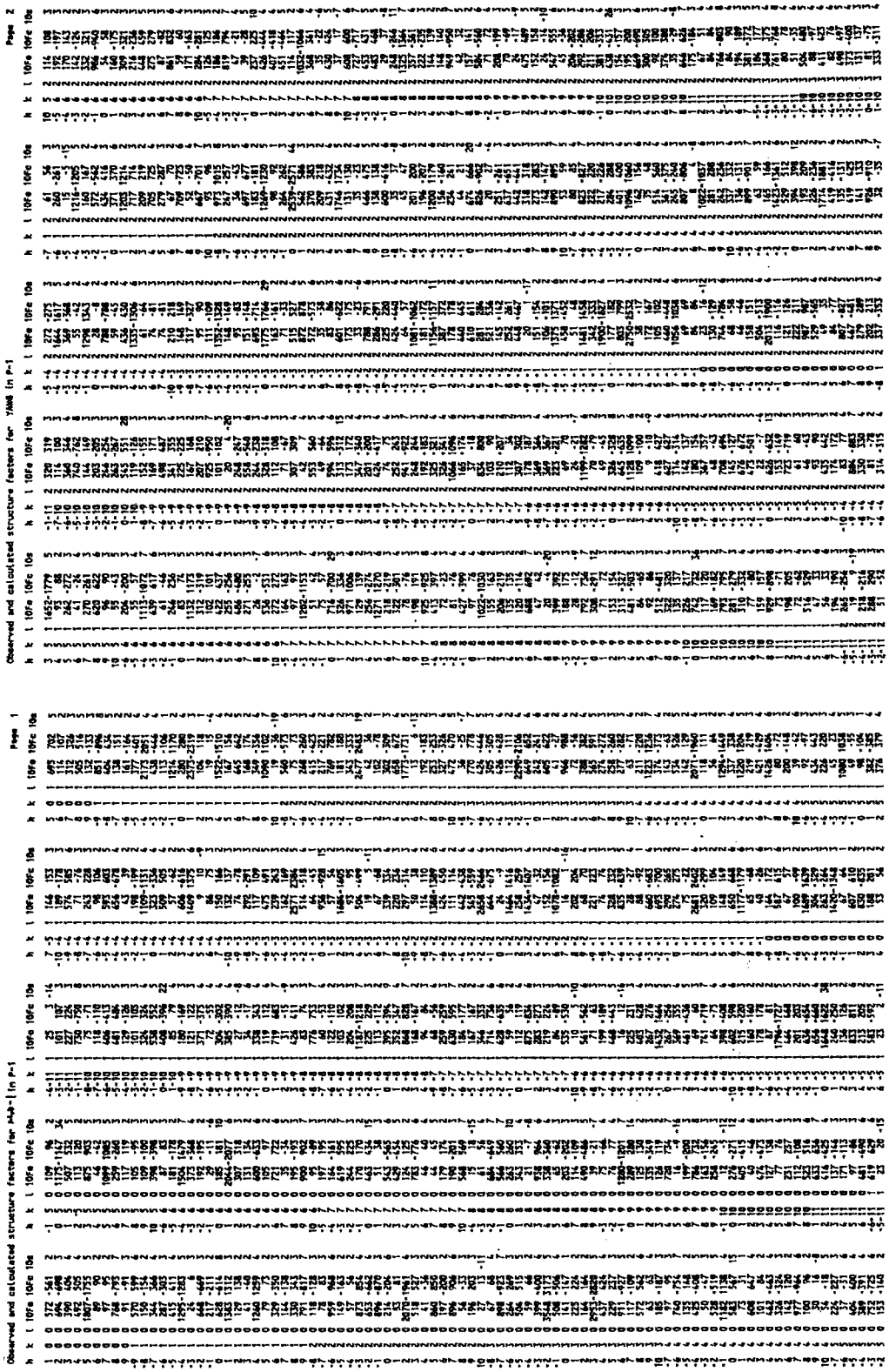
The anisotropic displacement exponent takes the form:

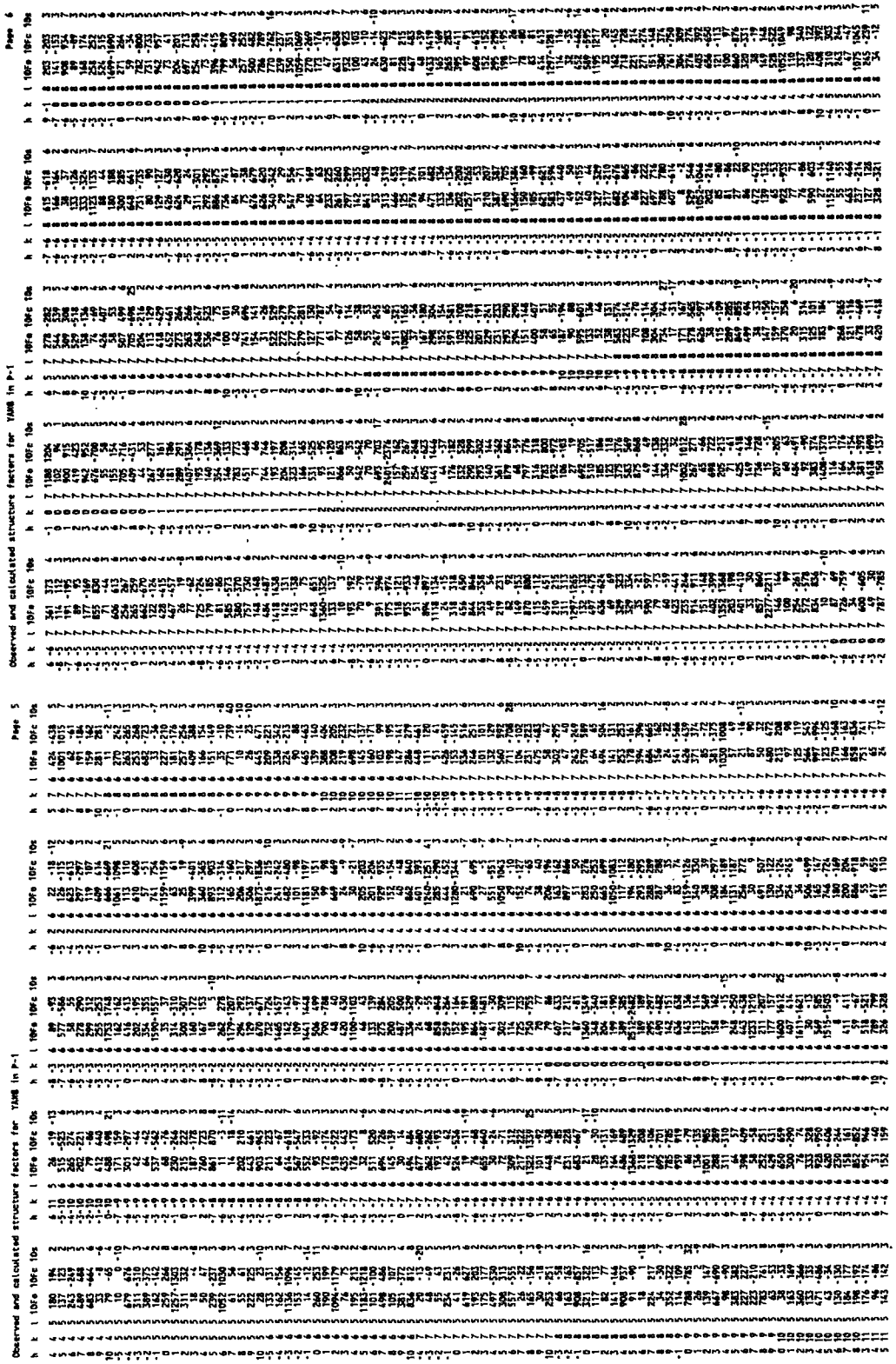
$-2\pi^2(h^2a^{*2}U_{11} + \ldots + 2hka^*b^*U_{12})$

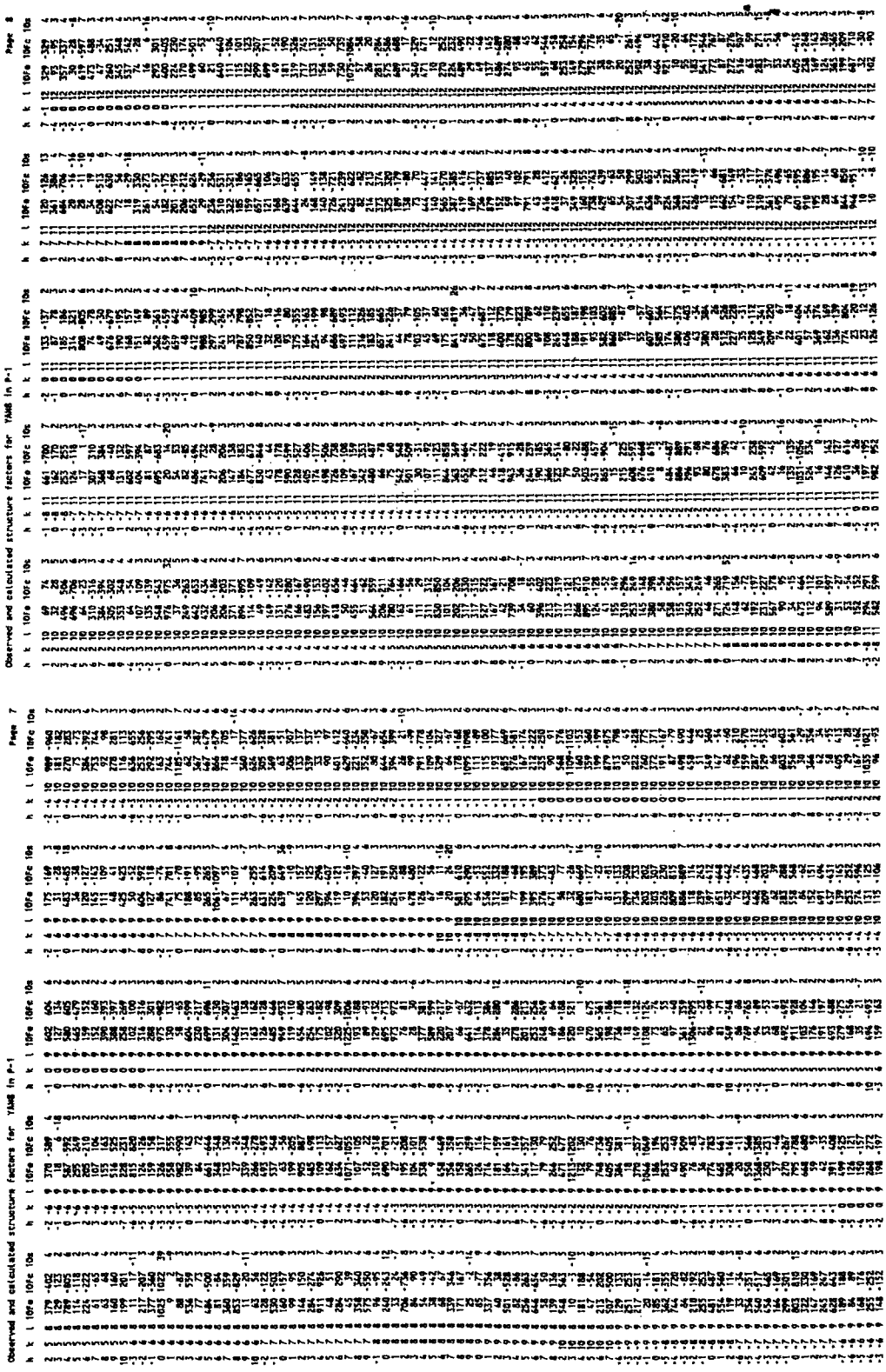
Table 5. H-Atom coordinates ($x10^4$) and isotropic displacement coefficients ($Å^2 x 10^3$)

	x	y	z	U
H(1A)	1814	5169	10727	50
H(1B)	3833	4286	10719	50
H(2A)	1198	3013	11548	50
H(2B)	3231	2127	11494	50
H(3A)	3910	1794	9645	50
H(3B)	1999	2972	9591	50
H(4A)	833	1116	10340	50
H(4B)	2673	-33	10523	50
H(5A)	2270	-1074	9028	40
H(5B)	3745	-377	8581	40
H(6A)	105	1069	8511	50
H(6B)	1373	2033	8250	50
H(1C)	2427	6069	12181	50
H(1D)	1123	5302	12566	50
H(1E)	3016	4443	12589	50
H(6C)	1220	976	6692	50
H(6D)	3071	249	6827	50
H(6E)	1890	-664	7068	50

I-818-12
MoO-1







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