PREPARATION of $[BzMe_3N]_2[Na_2W_4O_{12}(OMe)_4(MeOH)_6].6MeOH$

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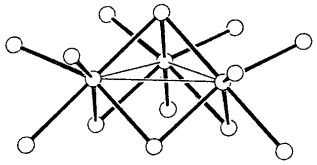
Abstract

An attempt to prepare trinuclear tungsten oxoalkoxides $[BzMe_3N]_3[W_3O_8(OMe)_5]$ from the reaction between $WO_2(OMe)_2$, $[BzMe_3N]_2WO_4$ and $[BzMe_3N](OMe)$ in the ratio of 2:1:1 in MeOH produced $[BzMe_3N]_2[Na_2W_4O_{12}(OMe)_4(MeOH)_6].6MeOH$. Suitable crystals for X-ray chrystallographic studies were obtained from hot mixture of methanol-acetonitrile solution. The I.R., ^{1}H-NMR, and microanalysis data including crystal structure of $[Na_2W_4O_{12}(OMe)_4(MeOH)_6]^{2-}$ anion are reported.

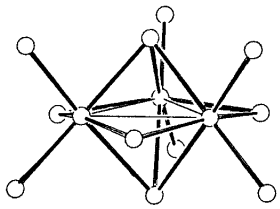
Keywords : sodium molybdenum oxoalkoxide, $[BzMe_3N]^+$ cation = $[(C_6H_5CH_2)N(CH_3)_3]^+$, X-ray crystallographic structure.

1. Introduction

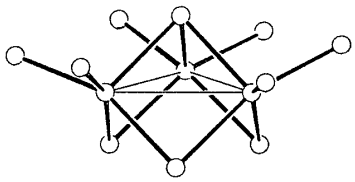
Recently a new class of triangulo-M_3 complexes of molybdenum and tungsten oxoalkoxides have been discovered. These clusters have either bicapped or hemicapped structures with μ_3-halides, –nitrogen, -oxygen atom or -alkoxide, with either M_3X_{13}, M_3X_{11} or M_3X_{10} skeletal geometries [1] as shown in Figure 1. These may or may not contain metal-metal bonding. One such example is $Mo_3O(OR)_{10}$ (where R= Pr^i, CH_2Bu^t) which was reported by Chisholm *et al*. [2,3] The compound has the M_3X_{11} sekeleton with two capped faces, (Mo_3-μ_3-O) and (Mo_3-μ_3-OR), and contains metal-metal bonding. Another kind of trinuclear with M_3X_{13} skeletal geometries has also been synthesized and structurally characterized by Bradley and his colleagues, [4] in $[P(CH_2Ph)Ph_3][W_3Cl_7(NBu^t)_3(\mu\text{-}NPh)_3]$. The $[W_3Cl_7(NBu^t)_3(\mu\text{-}NPh)_3]^-$ anion adopts the *triangulo*-M_3X_{13} type of structure, which has one capping chloro, (W_3-μ_3-Cl), three bridging imido groups, (W_2-μ-NPh), and does not contain any metal-metal bond. Here in this experiment we attempted to prepare a new *triangulo*-M_3 complexes of tungsten oxoalkoxide, $[BzMe_3N]_3[W_3O_8(OMe)_5]$ with M_3X_{13} skeletal geometries.



a.



b.



c.

Figure 1. (a). M_3X_{13}, (b). M_3X_{11}, (c). M_3X_{10} skeleton

2. Experimental

All the reactions and manipulations were carried out under an atmosphere of dry, oxygen-free nitrogen using Sclenk techniques with a nitrogen/vacuum manifold.

(i). Preparation of $WO_2(OMe)_2$.

WO_2Cl_2.dme (1 g, 2.65 mmol) and NaOMe (0.29 g, 5.31 mmol) were dissolved in MeOH (20 mL) and stirred overnight. The white solid that formed was removed by filtration. The solvent was removed in *vacuo* to give a thick colourless oil which was then washed with Et_2O (2x15 mL). All attempts to grow crystals were unsuccessful, but produced a white amorphous solid. Yield 0.43 g, 58.4 %. I.R. data: 365m, 570s, 845s(b), 890s(b), 980s, 1050s, 1160m cm^{-1}.

(ii). Preparation of $[BzMe_3N]_2[Na_2W_4O_{12}(OMe)_4(MeOH)_6]$.6MeOH

Solid $WO_2(OMe)_2$ (0.9 g, 3.24 mmol) and $[BzMe_3N]_2WO_4$ (0.89 g, 1.62 mmol) were suspended in the Schlenk tube with MeOH (15 mL). The solution of $[BzMe_3N](OMe)$ in MeOH (0.29 g, 1.62 mmol, 0.83 mL) was then added into the suspension and stirred overnight. The remaining solid was removed by filtration and the filtrate was stripped in *vacuo* to yield a white solid which was then washed with Et_2O (2x20 mL). The crystals of $[BzMe_3N]_2[Na_2W_4O_{12}(OMe)_4(MeOH)_6]$.6MeOH was grown in a hot mixture of MeCN-MeOH and allowed to cool to room temperature. I.R. data: 370s(b), 500m, 535m, 590w, 620s, 650s, 705s, 725s, 780m, 890s(b), 930s, 970s, 1030s, 1160w, 1215w, 1260w, 1590w, 1655w, 3250w(b) cm^{-1}. ^{1}H-NMR data: δ_H 7.8 (10H, m, C_6H_5-CH_2N), 5.1 (12H, s, ($CH_3O)_4$-W), 4.8 (4H, s, C_6H_5-CH_2N}, 3,5 (18H, m, ($CH_3OH)_3$-Na) and 3.3 ppm (18H, s, ($CH_3)_3$-N). Elemental analysis for $[BzMe_3N]_2$ $[Na_2W_4O_{12}(OMe)_4(MeOH)_6]$.6MeOH crystals found (calc.) %N 2.05 (2,02), %C 2.71 (2.65) and %H 6.73 (6,69).

3. Result and Discussions

An attempted preparation a *triangulo*- M_3X_{13} complex of tungsten oxoalkoxide, $[BzMe_3N]_3[W_3O_8(OMe)_5]$ by reacting $WO_2(OMe)_2$, $[BzMe_3N]_2WO_4$ and $[BzMe_3N]$ (OMe) with the ratio of 2:1:1 in MeOH produced colourless crystals of $[BzMe_3N]_2[Na_2W_4O_{12}(OMe)_4\ (MeOH)_6]$.6MeOH, **1**. The crystals have the characteristic I.R. bands at 370 cm^{-1} ($\nu_{O\text{-}W\text{-}O}$), 590, 620 cm^{-1} ($\nu_{W\text{-}OR}$), 890, 930 cm^{-1} ($\nu_{W=O}$), and 1030 cm^{-1} ($\nu_{C\text{-}O}$). The reaction seems not to proceed as might have been expected to proof $[BzMe_3N]_3[W_3O_8(OMe)_5]$, due to the ^{1}H-NMR spectrum which contains peaks at 7.8 (10H, m, C_6H_5-CH_2N), 5.1 (12H, s, $(CH_3O)_4$-W), 4.8 (4H, s, -CH_2N), 3,5 (18H, m, $(CH_3OH)_3$-Na) and 3.3 ppm (18H, s, $(CH_3)_3N$) in the ratio of 5:6:2:9:9 which is agree for the formula of $[BzMe_3N]_2[Na_2W_4O_{12}(OMe)_4\ (MeOH)_6]$.6MeOH, **1**. Also the microanalysis fits to compound **1** instead of that expected formula, $[BzMe_3N]_3[W_3O_8\ (OMe)_5]$. The crystals for X-ray crystallographic studies were grown from hot solution mixture of MeCN-MeOH. The crystal structure of anion **1**, $[Na_2W_4O_{12}(OMe)_4\ (MeOH)_6]^{2-}$ is illustrated in Figure 2 and the selected bond lengths and angles displayed in Table 1 (see Apendix 1).

The molecular structure of **1** contains two sodium atoms, suggesting that the $WO_2(OMe)_2$ starting material produced from reaction between WO_2Cl_2.dme and NaOMe has a formula of $WO_2(OMe)_2$.xNaOMe. The product has similar features in the IR spectrum to those described for $WO_2(OMe)_2$ which is produced from the reaction of WO_2Cl_2 with NaOMe by Kucheiko and Turova [5]. Therefore $WO_2(OMe)_2$.xNaOMe has not yet been fully characterized.

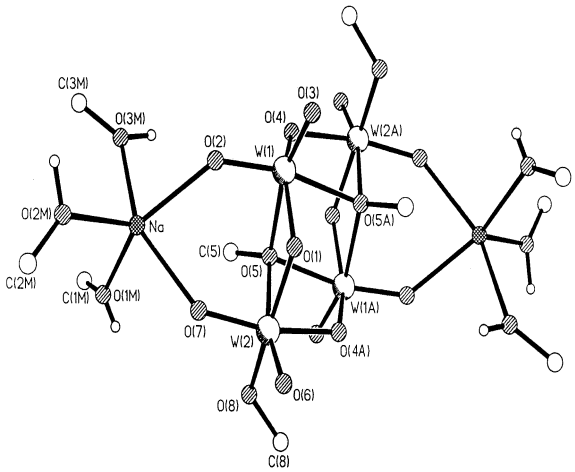


Figure 2. The structure of $[Na_2W_4O_{12}(OMe)_4(MeOH)_6]^{2-}$ anion

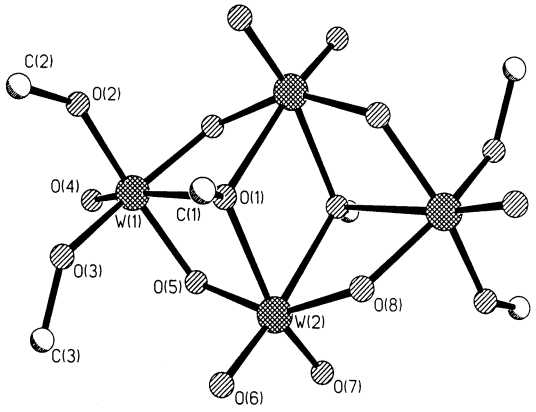


Figure 3. The tetra nuclear unit of $[W_4O_8(\mu\text{-}O)_4(OMe)_2(\mu_3\text{-}OMe)_2]^{4-}$ structure.

As can be seen in Figure 2, the overall crystal structure of **1** contains four octahedral $[W_4O_8(\mu\text{-}O)_4(OMe)_2(\mu_3\text{-}OMe)_2]$ and two distored trigonal bipyramidal of $[NaO_2(MeOH)_3]^{3-}$ which is connected each other by corner-sharing through oxygen atoms. The four distorted octahedral of $[W_4O_8(\mu\text{-}O)_4(OMe)_2(\mu_3\text{-}OMe)_2]^{4-}$ structure (Figure 3) consists of compact cluster of four edge-sharing octahedral and has similar structure to those described earlier by Havelock [6] in her structure of $[Pr^n{}_4N]_2[W_4O_{10}(OMe)_6]$ and also by Zubieta and co-workers [7] in their compound of $[Ph_3MeP]_2[Mo_4O_{10}(OMe)_6]$.

As seen in Figure 2, the two distorted trigonal bipyramidal of $[NaO_2(MeOH)_3]^{3-}$ is connected to tetra nuclear unit of $[W_4O_8(\mu\text{-}O)_4(OMe)_2\ (\mu_3\text{-}OMe)_2]^{4-}$ through two oxygen atoms each at O_2 and O_7, and O_{2a} and O_{7a}. The trigonal bipyramidal coordination geometry adopted by $[NaO_2(MeOH)_3]^{3-}$ is very unusual coordination, since the Na atom bond to three MeOH molecule through oxygen atom which is still coordinated with hydrogen atom, and this is the first structure been reported.

4. Conclusion

The preparation a new complex oxo alkoxide with M_3X_{13} skeletal geometries of $[BzMe_3N]_3[W_3O_8\ (OMe)_5]$ has not been successful. However, the reaction produced a novel compound $[BzMe_3N]_2[Na_2W_4O_{12}\text{-}(OMe)_4(MeOH)_6].6MeOH$. This might be due to the WO_2Cl_2.dme was used instead of WO_2Cl_2, resulting in formation of $WO_2(OMe)_2$.xNaOMe which has not been expected.

Acknowledgement

Author would like to express gratitude to R.J. Errington, W. Clegg and Co-workers, also D. Dunbar at the University of Newcastle upon Tyne, UK, for valuable discussions and help during the course of this work. Author also thank the World Bank Project for their support of this work.

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Appendix 1.

Table 1. Selected Bond Length (Å)and Angles (deg) for $[BzMe_3N]_2[Na_2W_4O_{12}(OMe)_4(MeOH)_6]$.6MeOH.

W1-O1	1.921(5)	W2-O1	1.940(5)
W1-O2	1.739(5)	W2-O4a	2.068(5)
W1-O3	1.733(5)	W2-O5	2.350(5)
W1-O4	1.893(5)	W2-O6	1.724(5)
W1-O5	2.311(5)	W2-O7	1.754(5)
W1-O5a	2.251(5)	W2-O8	1.971(5)
W1a-O5	2.251(5)	W2a-O4	2.068(5)
Na-O1m	2.316(7)	Na-O3m	2.396(8)
Na-O2M	2.282(7)	Na-O7	2.386(6)

O(1)-W(1)-O(2)	99.6(2)	O(1)-W(2)-O(4a)	83.8(2)
O(1)-W(1)-O(3)	99.6(2)	O(1)-W(2)-O(5)	72.3(2)
O(1)-W(1)-O(4)	149.6(2)	O(1)-W(2)-O(6)	98.3(2)
O(1)-W(1)-O(5)	73.5(2)	O(1)-W(2)-O(7)	95.8(2)
O(1)-W(1)-O(5a)	80.9(2)	O(1)-W(2)-O(8)	159.5(2)
O(2)-W(1)-O(3)	104.8(2)	O(4a)-W(2)-O(5)	69.1(2)
O(2)-W(1)-O(4)	98.4(2)	O(4a)-W(2)-O(6)	99.2(2)
O(2)-W(1)-O(5)	89.4(2)	O(4a)-W(2)-O(7)	154.3(2)
O(2)-W(1)-O(5a)	161.1(2)	O(4a)-W(2)-O(8)	83.6(2)
O(3)-W(1)-O(4)	102.3(2)	O(5)-W(2)-O(6)	165.4(2)
O(3)-W(1)-O(5)	164.1(2)	O(5)-W(2)-O(7)	86.2(2)
O(3)-W(1)-O(5a)	93.9(2)	O(5)-W(2)-O(8)	88.1(2)
O(4)-W(1)-O(5)	82.3(2)	O(6)-W(2)-O(7)	106.3(2)
O(4)-W(1)-O(5a)	74.3(2)	O(6)-W(2)-O(8)	99.6(2)
O(5)-W(1)-O(5a)	72.5(2)	O(7)-W(2)-O(8)	88.6(2)

W1-O1-W2	119.6(2)	W1a-O5-W1	107.5(2)
W1-O4-W2a	119.5(3)	W1a-O5-W2	96.1(2)
W1-O5-W2	91.4(2)		
W1-O2-Na	138.0(3)	W2-O7-Na	141.4(3)

O1m-Na-O2	139.7(2)	O2-Na-O3m	77.1(2)
O1m-Na-O2m	101.2(3)	O2-Na-O7	82.2(2)
O1m-Na-O3m	92.7(3)	O2m-Na-O3m	99.9(3)
O1m-Na-O7	87.8(2)	O2m-Na-O7	110.9(3)
O2-Na-O2m	118.9(2)	O3m-Na-O7	148.5(2)

Table 2. Crystal data, structure solution and refinement for rb-37

Identification code	rb-37
Chemical formula	$C_{36}H_{92}N_2Na_2O_{28}W_4$
Formula weight	1782.50
Temperature	160(2) K
Radiation and wavelength	MoKα, 0.71073 A
Crystal system, space group	triclinic, P1
Unit cell dimensions	a - 11.042(9) Å α = 32.77(4)°
	b - 11.189(9) Å β = 77.lO(5)°
	c - 13.745(12) Å γ = 67.88(5)°
Volume	1532(2) $Å^3$
Z 1	
Density (calculated)	1.932 g/cm^3
Absorption coefficient μ	7.576 mm^{-1}
F(000)	864
Reflections for cell refinement	29 (θ range 11.27 to 12.52°)
Crystal colour	colourless
Crystal size	.35 x .31 x .15 mm
Data collection method	Stoe-Siemens diffractometer, ω / θ scans
θ range for data collection	2.54 to 22.50°
Index ranges	$-11 \leq h \leq 11$, $-11 \leq$ I: ≤ 12, $-14 \leq \ell \leq 14$
Standard reflections	5 every 60 minutes
Intensity decay of standards	5%
Reflections collected	5806
Independent reflections	4002 (R_{int} = 0.0207)
Reflections with I>2σ (I)	3472
Absorption correction	empirical (SHELXA)
Max. and min. transmission	.46184 and .16208
Structure solution	direct methods
Refinement method	full—matrix least—squares on F^2
Weighting parameters a, b	0.0656, 1.9752
Data / restraints / parameters	4001 / 2 / 347
Goodness—of—fit on F^2	1.072
Final R indices [I>2 σ (I)]	R1 = 0.0327, wR2 = 0.0864
R indices (all data)	R1 = 0.0399, wR2 = 0.0915
Largest and mean shift/esd	- 0.001 and 0.000
Largest diff. peak and hole	2.820 and —2.362 $eÅ^{-3}$

Symmetry transformations used to generate equivalent atoms:
A: -x+l, -y+2, -z

Table 3. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for rb-37 . U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
W(1)	4626.1(3)	8527.9(2)	16.2(2)	13.17(13)
W(2)	7748.3(3)	8458.4(2)	-423.9(2)	14.79(13)
0(1)	6322(4)	7979(4)	414(4)	16.3(11)
0(2)	5063(5	7564(5)	-998(4)	18.4(l1)
0(3)	3890(5)	7736(4)	993(4)	21.4(12)
0(4)	3133(4)	9874(4)	-366(4)	15.5(11)
0(5)	5747(4)	9788(4)	-928(3)	14.2(10)
C(S)	5627(7)	10099(7)	-1952(5)	17(2)
0(6)	8989(5)	7616(5)	242(4)	24.5(12)
0(7)	7988(5)	7500(5)	-1417(4)	18.6(11)
0(8)	8644 (5)	9494(5)	-1356(4)	22.3(12)
C(8)	9106(7)	10436(7)	-1119(7)	27(2)
N	12418(6)	5860(5)	-757(5)	22.5(14)
C(9)	13877(8)	5210(7)	-1146(7)	34(2)
C(10)	12177(9)	5768(8)	- 355(6)	32(2)
C(11)	12015(8)	7245(6)	-1113(6)	25(2)
C(12)	11610(7)	5204(7)	-1092(6)	24(2)
C(13)	11830(7)	5101(7)	-2197(6)	26(2)
C(14)	11064(8)	6034(8)	-2792(6)	30(2)
C(l5)	11266(9)	5920(8)	-3802(7)	41(2)
C(16)	12258(9)	4829(9)	-4252(7)	43(2)
C(17)	13016(10)	3874(9)	-3675(7)	43(2)
C(18)	12820(9)	3999(7)	-2654(7)	35(2)
Na	6778(3)	6972(3)	-2428(2)	26.5(7)
0(1M)	7809(7)	7928(7)	-3780(5)	43(2)
C(1M)	7322(17)	8581(16)	-4610(9)	112(7)
0(2M)	7746(6)	4851(6)	-2811(5)	48(2)
C(2M)	9082(11)	4021(12)	-3047(10)	66(3)
O(3M)	4795(7)	7569(7)	-3101(5)	49(2)
C(3M)	4363(10)	7217(12)	-3863(8)	62(3)
0(4M)	6369(7)	3330(6)	-2850(5)	49(2)
C(4M)	6796(11)	2510(9)	-3658(7)	48(3)
0(SM)	9168(6)	9268(6)	-3313(5)	34.0(14)
0(5K)	10397(9)	9422(9)	-3694(7)	46(2)
0(6M)	2701(5)	9630(5)	-2171(4)	27.7(13)
C(6M)	2386(10)	10901(8)	-2634(7)	39(2)

Table 4. Bond lengths (Å) and angles (°) for rb.37

W(l)-0(3)	1.733(5)	W(1)-O(2)	1.739(5)
W(l)-0(4)	1.893(5)	W(1)-0(1)	1.921(5)
W(1)-0(5a)	2.251(5)	W(1)-0(5)	2.311(5)
W(2)-0(6)	1.724(5)	W(2)-0(7)	1.754(5)
W(2)-0(l)	1.940(5)	W(2)-0(8)	1.971(5)
W(2)-0(4a)	2.068(5)	W(2)-0(5)	2.350(5)
0(2)-Na	2.369(6)	0(4)-W(2a)	2.068(5)
0(5)-C(S)	1.431(8)	0(5)-W(1a)	2.251(5)
0(7)-Na	2.386(6)	0(8)-C(8)	1.429(9)
N-C(10)	1.489(10)	N-C(11)	1.493(9)
N-C(9)	1.497(9)	N-C(12)	1.518(9)
C(12)-C(13)	1.496(11)	C(13)-C(14)	1.380(12)
C(l3)-C(18)	1.409(11)	C(14)-C(15)	1.370(12)
C(15)-C(l6)	1.393(12)	C(16)-C(17)	1.371(13)
C(17)-C(18)	1.389(13)	Na-0(2M)	2.282(7)
Na-O(1M)	2.316(7)	Na-0(3M)	2.396(8)
0(1M)-C(1M)	1.376(13)	0(2M)-C(2M)	1.405(12)
0(3M)-C(3M)	1.396(11)	O(4M)-C(4M)	1.412(11)
0(5M)-C(5M)	1.407(10)	C(6M)-C(6M)	1.431(10)
0(3)-W(1)-0(2)	104.8(2)	0(3)-W(1)-0(4)	102.3(2)
0(2)-W(1)-0(4)	98.4(2)	0(3)-W(l)-0(1)	96.6(2)
0(2)-W(1)-0(1)	99.6(2)	0(4)-W(1)-0(1)	149.6(2)
0(3)-W(l)-0(5a)	93.9(2)	O(2)-W(1)-0(5a)	161.1(2)
0(4)-W(1)-0(5a)	74.3(2)	0(l)-W(1)-0(5a)	80.9(2)
0(3)-W(1)-0(5)	164.1(2)	0(2)-W(1)-0(5)	89.4(2)
0(4)-W(1)-0(5)	82.3(2)	0(1)-W(1)-0(5)	73.5(2)
0(Sa)-W(1)-0(5)	72.5(2)	0(6)-W(2)-0(7)	106.3(2)
0(6)-W(2)-0(1)	98.3(2)	0(7)-W(2)-0(1)	95.8(2)
0(6)-W(2)-0(8)	99.6(2)	0(7)-W(2)-0(8)	88.6(2)
0(1)-W(2)-0(8)	159.5(2)	0(6)-W(2)-0(4a)	99.2(2)
0(7)-W(2)-0(4a)	154.3(2)	0(1)-W(2)-0(4a)	83.8(2)
0(8)-W(2)-0(4a)	83.6(2)	0(6)-W(2)-0(5)	165.4(2)
0(7)-W(2)-0(5)	86.2(2)	0(1)-W(2)-0(5)	72.3(2)
0(8)-W(2)-0(5)	88.1(2)	0(4a)-W(2)-0(5)	69.1(2)
W(l)-0(l)-W(2)	119.6(2)	W(1)..O(2)-Na	138.0(3)
W(1)-0(4)-W(2a)	119.5(3)	C(5)-O(5)-W(la)	115.5(4)
C(5)-0(5)-W(1)	118.7(4)	W(1a)-0(5)-W(1)	107.5(2)
C(5)-0(5)-W(2)	123.3(4)	W(la)-0(5)-W(2)	96.1(2)
W(l)-0(5)-W(2)	91.4(2)	W(2)-0(7)-Na	141.4(3)
C(8)-0(8)-W(2)	127.8(5)	C(l0)-N.C(11)	109.6(6)
C(10)-N-C(9)	108.6(6)	C(11)-N-C(9)	108.3(6)
C(10)-N-C(12)	107.9(6)	C(11)-N-C(12)	111.1(6)
C(9)-N-C(12)	111.3(6)	C(13)-C(12)-N	115.1(6)
C(14)-C(13)-C(18)	118.0(8)	C(14)-C(13)-C(12)	122.3(7)
C(18)-C(13)-C(12)	119.7(7)	C(15)-C(14)-C(13)	121.7(8)
C(14)-C(15)-C(16)	119.9(8)	C(17)-C(16)-C(15)	119.6(9)
C(16)-C(17)-C(1S)	120.4(8)	C(17)-C(18)-C(13)	120.4(8)
0(2M)-Na-0(1M)	101.2(3)	0(2M)-Na-0(2)	118.9(2)
0(1M)-Na-0(2)	139.7(2)	0(2M)-Na-0(7)	110.9(3)
0(1M)-Na-0(7)	87.8(2)	0(2)-Na-0(7)	82.2(2)
0(2M)-Na-0(3M)	99.9(3)	0(1M)-Na-0(3M)	92.7(3)

0(2)-Na-0(3M)	77.1(2)	0(7)-Na-0(3M)	148.5(2)
C(1M)-0(1M)-Na	128.7(7)	C(2M)-0(2M)-Na	132.5(6)
C(3M)-0(3M)-Na	139.0(6)		

Table 5. Anisotropic displacement parameter.3 ($Å^2$ x 10^3) for rb-37. The anisotropic displacement factor exponent takes the form: $-2\pi^2(h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12})$.

	U(11)	U(22)	U(33)	U(23)	U(13)	11(12)
W(1)	11.6(2)	6.9(2)	19.7(2)	-0.64(13)	-0.12(13)	-3.49(13)
W(2)	10.7(2)	8.7(2)	22.2(2)	-1.50(13)	0.00(14)	-1.94(14)
0(1)	14(3)	5(2)	27(3)	1(2)	-3(2)	-1(2)
0(2)	19(3)	17(3)	22(3)	0(2)	-5(2)	-10(2)
0(3)	18(3)	16(3)	29(3)	1(2)	-4(2)	-5(2)
0(4)	9(2)	13(2)	22(3)	-3(2)	1(2)	-2(2)
0(5)	12(3)	13(2)	18(3)	0(2)	-2(2)	-6(2)
C(S)	17(4)	15(4)	19(4)	4(3)	-2(3)	-8(3)
0(6)	12(3)	20(3)	36(3)	-2(2)	-1(2)	-1(2)
0(7)	14(3)	17(3)	22(3)	-3(2)	2(2)	-5(2)
0(8)	21(3)	18(3)	30(3)	-3(2)	2(2)	-13(2)
C(8)	13(4)	16(4)	53(6)	-7(4)	-5(4)	-5(3)
N	18(3)	12(3)	33(4)	5(3)	-4(3)	-3(3)
C(9)	19(4)	16(4)	64(6)	-5(4)	-5(4)	-2(3)
C(1O)	40(5)	23(4)	36(5)	5(4)	-16(4)	-14(4)
C(ll)	24(4)	9(4)	41(5)	-2(3)	-3(4)	-5(3)
C(12)	24(4)	17(4)	32(5)	-2(3)	2(4)	-11(3)
C(13)	16(4)	20(4)	46(5)	-2(4)	-1(4)	-13(3)
C(l4)	24(4)	30(5)	35(5)	-4(4)	-4(4)	-7(4)
C(15)	36(5)	34(5)	44(6)	6(4)	-12(4)	-4(4)
C(16)	45(6)	45(6)	31(5)	-9(4)	2(4)	-11(5)
C(17)	45(6)	38(5)	36(6)	-14(4)	-3(5)	-5(4)
C(18)	38(5)	13(4)	52(6)	-2(4)	-11(4)	-3(4)
Na	29(2)	22(2)	28(2)	-7.2(13)	2.3(13)	-10.3(13)
0(1M)	56(5)	54(4)	33(4)	-7(3)	3(3)	-40(4)
C(lM)	203(18)	180(16)	44(8)	59(9)	-72(10)	-163(15)
0(2M)	46(4)	27(3)	73(5)	-14(3)	3(3)	-19(3)
C(2M)	46(7)	68(8)	88(9)	-21(7)	-3(6)	-19(6)
0(3M)	35(4)	63(5)	47(4)	-25(3)	-6(3)	-11(3)
C(3M)	52(7)	98(9)	40(6)	-32(6)	3(5)	-31(6)
0(4M)	68(5)	45(4)	39(4)	2(3)	-2(3)	-32(4)
C(4M)	68(7)	44(6)	33(5)	2(4)	0(5)	-30(5)
0(5M)	36(3)	47(4)	27(3)	0(3)	-1(3)	-27(3)
C(5M)	50(6)	51(6)	44(6)	3(5)	1(5)	-32(5)
0(6M)	32(3)	24(3)	29(3)	2(2)	-7(3)	-13(2)
C(6M)	50(6)	36(5)	35(5)	11(4)	-16(4)	-20(5)

Table 6. Hydrogen atom coordinates (x lO^4) and isotropic displacement parameters (Å ^{2} x 10^3) for rb-37.

	x	y	z	U
H(5A)	5806(7)	9308(7)	-2285(5)	26
H(5B)	6269(7)	10502(7)	-2285(5)	26
H(5C)	4722(7)	10699(7)	-1989(5)	26
H(8A)	9511(7)	10794(7)	-1736(7)	41
H(8B)	9770(7)	10028(7)	-693(7)	41
H(8C)	8354(7)	11131(7)	-764(7)	41
H(9A)	14049(8)	5263(7)	-1878(7)	52
H(9B)	14163(8)	4301(7)	-909(7)	52
H(9C)	14373(8)	5639(7)	-905(7)	52
H(1OA)	12442(9)	4857(8)	537(6)	47
H(1OB)	11228(9)	6214(8)	620(6)	47
H(1OC)	12702(9)	6171(8)	589(6)	47
H(11A)	12174(8)	7304(6)	-1845(6)	38
H(11B)	12540(8)	7649(6)	-879(6)	38
H(11C)	11066(8)	7693(6)	-849(6)	38
H(12A)	11827(7)	4324(7)	-773(6)	29
H(12B)	10653(7)	5689(7)	-845(6)	29
H(14)	10379(8)	6775(8)	-2494(6)	36
H(15)	10733(9)	6583(8)	-4196(7)	49
H(16)	12405(9)	4749(9)	-4952(7)	51
H(17)	13678(10)	3123(9)	-3976(7)	51
H(18)	13357(9)	3337(7)	-2262(7)	42
H(1M)	8283(114)	8223(111)	-3674(86)	65
H(1M1)	7996(17)	8874(16)	-5051(9)	168
H(1M2)	6516(17)	9330(16)	-4403(9)	168
H(1M3)	7107(17)	8005(16)	-4967(9)	168
H(2M)	6996(108)	4432(103)	-2724(81)	72
H(2M1)	9335(27)	3907(65)	-3770(13)	102
H(2M2)	9189(20)	3181(29)	-2694(50)	102
H(2M3)	9653(13)	4397(40)	-2844(58)	102
H(3M1)	3426(10)	7753(12)	-3856(8)	92
H(3M2)	4458(10)	6306(12)	-3758(8)	92
H(3M3)	4902(10)	7346(12)	-4509(8)	92
H(3M)	4270(128)	8356(127)	-2814(98)	92
H(4M1)	6955(63)	3001(18)	-4234(8)	71
H(4M2)	6107(29)	2166(50)	-3677(30)	71
H(4M3)	7622(36)	1795(35)	-3573(26)	71
H(4M)	6533(101)	2819(24)	-2271(15)	71
H(5M1)	10809(31)	8980(53)	-4323(25)	69
H(5M2)	10987(24)	9049(56)	-3209(21)	69
H(5M3)	10252(12)	10343(10)	-3815(46)	69
H(5M)	9016(113)	9154(109)	-2689(87)	69
H(6M1)	1789(48)	11016(21)	-3097(33)	59
H(6M2)	3207(11)	11016(21)	-3001(37)	59
H(6M3)	1947(54)	11541(8)	-2120(8)	59
H(6M)	2668(99)	9690(96)	-1496(82)	59