Apéndice

1. Apéndice I. Síntesis de los Catalizadores

Cantidades de reactivos usadas para la síntesis de los catalizadores

Muestra	$C_{10}H_{14}O_5V$ (gr)	$(C_5H_8O_2)_3Mn$ (gr)	Metanol (ml)
MnO_x(ac)	--	6,40	100
MnV12(ac)	0,90	8,81	150
MnV20(ac)	1,66	8,81	165
MnV30(ac)	2,18	6,77	140

Muestra	$NaVO_3$ (gr)	NaOH (gr)	$NaMnO_4$ (gr)	Etanol (ml)
MnO_x(Na)	--	0,23	3,20	2,0
MnV12(Na)	0,33	0,23	3,20	2,5
MnV20(Na)	0,81	0,23	3,20	2,5
MnV30(Na)	1,22	0,12	1,6	1,1

Muestra	KVO_3 (gr)	KOH (gr)	$KMnO_4$ (gr)	Etanol (ml)
MnO_x(K)	--	0,23	3,16	2,0
MnV12(K)	0,38	0,23	3,16	2,5
MnV20(K)	0,92	0,23	3,16	2,5
MnV30(K)	1,14	0,11	1,44	1,1

Muestra	$NaVO_3$ (gr)	NaOH (gr)	$KMnO_4$ (gr)	Etanol (ml)
MnO_x(NaK)		0,12	1,58	1,5
MnV12(NaK)	0,32	0,12	2,63	1,2
MnV20(NaK)	0,40	0,12	1,58	1,1
MnV30(NaK)	1,00	0,12	1,44	1,1

2. **Apéndice II.** Calibraciones para TPSR

2.1 Calibración de Metanol

Se prepararon soluciones de distintas concentraciones de metanol (Carlo Erba 99,9 %) en agua destilada. Estas soluciones fueron:

Me0,5: 0,5 ml de metanol + 9,5 ml de agua

Me1: 1 ml de metanol + 9 ml de agua

Me1,5: 1,5 ml de metanol + 8,5 ml de agua

Me2: 2 ml de metanol + 8 ml de agua

Me3: 3 ml de metanol + 7 ml de agua

Me4: 4 ml de metanol + 6 ml de agua

Se inyectó en el espectrómetro de masas y 0,5 µl de cada una de ellas, y se graficó. Los valores de área son un promedio de tres medidas.

Solución	Área	µmoles
Me0,5	751	0,62
Me1	1542	1,23
Me1,5	2072	1,85
Me2	3523	2,47
Me3	4133	3,70
Me4	5093	4,94

La curva de calibración responde a la siguiente forma:

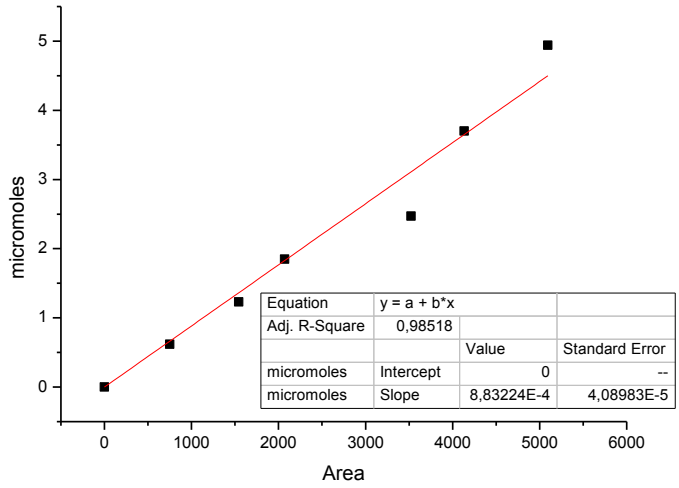


Figura 1: Curva de calibración para metanol.

2.2 Calibración de formaldehído

El formol no se comercializa como una droga pura, sino en soluciones de 32 a 37 % p/v estabilizadas con metanol para evitar que polimericen fundamentalmente a paraformaldehído. Se valoró el formaldehido por el método del sulfito de sodio y se prepararon soluciones de HCHO en metanol.

Esta metodología que no es interferida por la presencia de alcoholes, se fundamenta en la liberación cuantitativa de hidróxido de sodio cuando el formaldehido y el sulfito de sodio reaccionan para dar un producto de adición:

$$HCHO + Na_2SO_3 + H_2O \rightarrow NaOH + CH_2(NaSO_3)OH$$

El hidróxido se valoró con un ácido de concentración conocida, en presencia de un indicador.

Como en el caso anterior se obtuvieron las áreas de las señales cromatográficas por inyección de las soluciones preparadas.

Se colocó cantidades variables de formaldehído (11,68 M) en un matraz de 10 ml y se enrazó con metanol obteniendo diferentes soluciones:

F0,5: 0,5 ml de formaldehído (molaridad de la sol formada: 0,584)

F1: 1 ml de formaldehído (molaridad de la solución formada: 1,168)

F1,5: 1,5 ml de formaldehído (molaridad de la solución formada:1,752)

F2: 2 ml de formaldehído (molaridad de la solución formada: 2,336)

F2,5: 2,5 ml de formaldehído(molaridad de la solución formada:2,921)

F3: 3 ml de formaldehído (molaridad de la solución formada: 3,505)

F4: 4 ml de formaldehído (molaridad de la solución formada: 4,672)

Muestra	Area	µmoles
F0,5	201	0,292
F1	597	0,584
F1,5	911	0,876
F2	1202	1,168
F2,5	1951	1,461
F3	2370	1,752
F4	2761	2,336

Graficando:

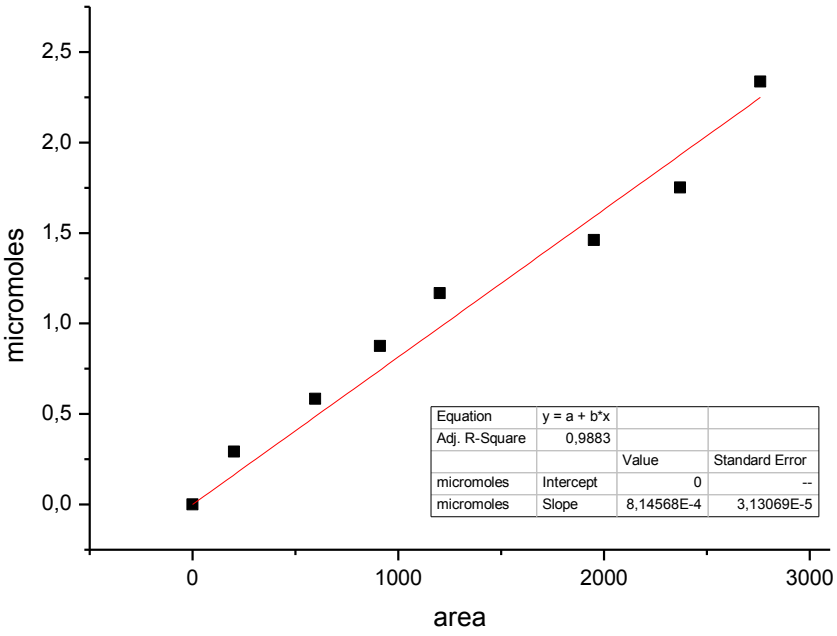
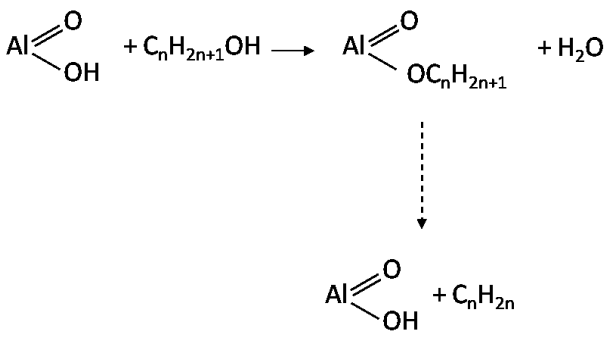


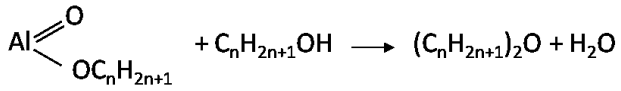
Figura 2: Curva de calibración para el formaldehído.

2.3 Calibración de dimetiléter

Debido a que no se dispone de cilindros de este gas, fue necesario sintetizarlo en el laboratorio por deshidratación de metanol sobre γ-Al_2O_3 en corriente de nitrógeno desde 150 a 280°C.

Según estudios realizados por Ipatieff (1), el mecanismo de deshidratación de alcoholes sobre alúmina involucra dos reacciones:





Primeramente el catalizador se secó en corriente de aire a 350° C. luego se hizo reaccionar con una corriente de 8 % moles de metanol en nitrógeno, a distintas temperaturas (100 a 280°C), con lo que se obtuvieron distintas conversiones de metanol.

En todos los casos el único producto de reacción fue dimetileter, según se pudo verificar por espectrometría de masas.

Los moles de éter generados se calcularon como la mitad de los moles convertidos de metanol. Los datos obtenidos se presentan a continuación.

Temperatura (°C)	Volumen de inyección (ml)	Masa	Área	µmoles
100	0,7	31	2441	3,35
	0,7	45	--	--
	1,0	31	5426	4,79
	1,0	45	--	--
150	0,7	31	2884	2,55
	0,7	45	--	--
	1,0	31	4227	--
	1,0	45	--	--
200	0,7	31	1957	1,73
	0,7	45	642	0,81
	1,0	31	2620	2,31
	1,0	45	822	1,24
250	0,6	31	564	0,47
	0,6	45	1141	1,2
	0,8	31	766	0,68
	0,8	45	1439	1,6
	1,0	31	899	0,79
	1,0	45	1629	2,00
280	0,7	31	452	0,4
	0,7	45	1135	1,48
	1,0	31	668	0,59
	1,0	45	1648	2,1

Y la gráfica resultante fue:

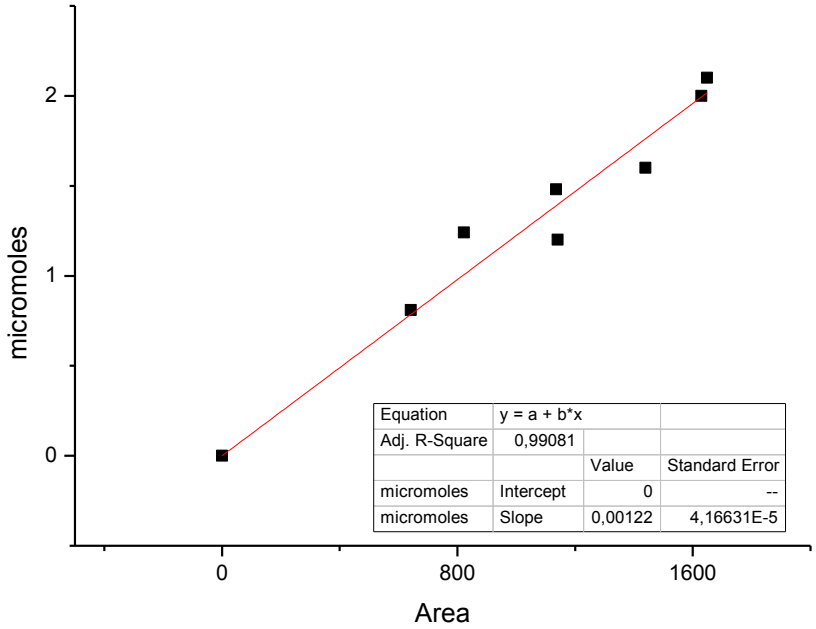


Figura 3: Curva de calibración para el dimetiléter.

2.4 Calibración de CO_2

La calibración se realizó inyectando en el espectrómetro de masas distintos volúmenes de CO_2.

Cantidad (ml)	Área	µmoles
0,1	7416	4,46
0,2	13229	8,93
0,3	20373	13,39
0,4	26648	17,86
0,5	31144	22,32

La regresión lineal de la curva de calibración representada por el área obtenida vs los micromoles de CO_2 inyectados. Se presenta a continuación:

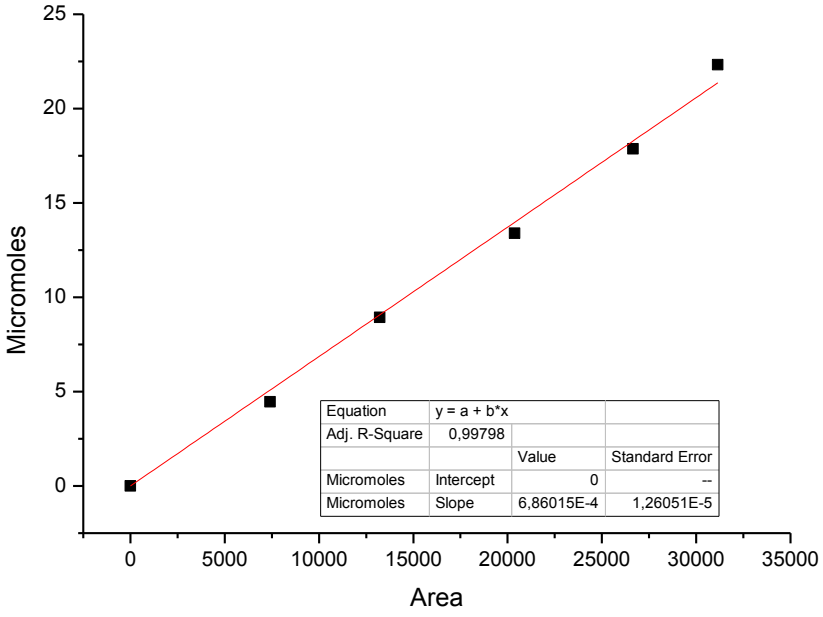
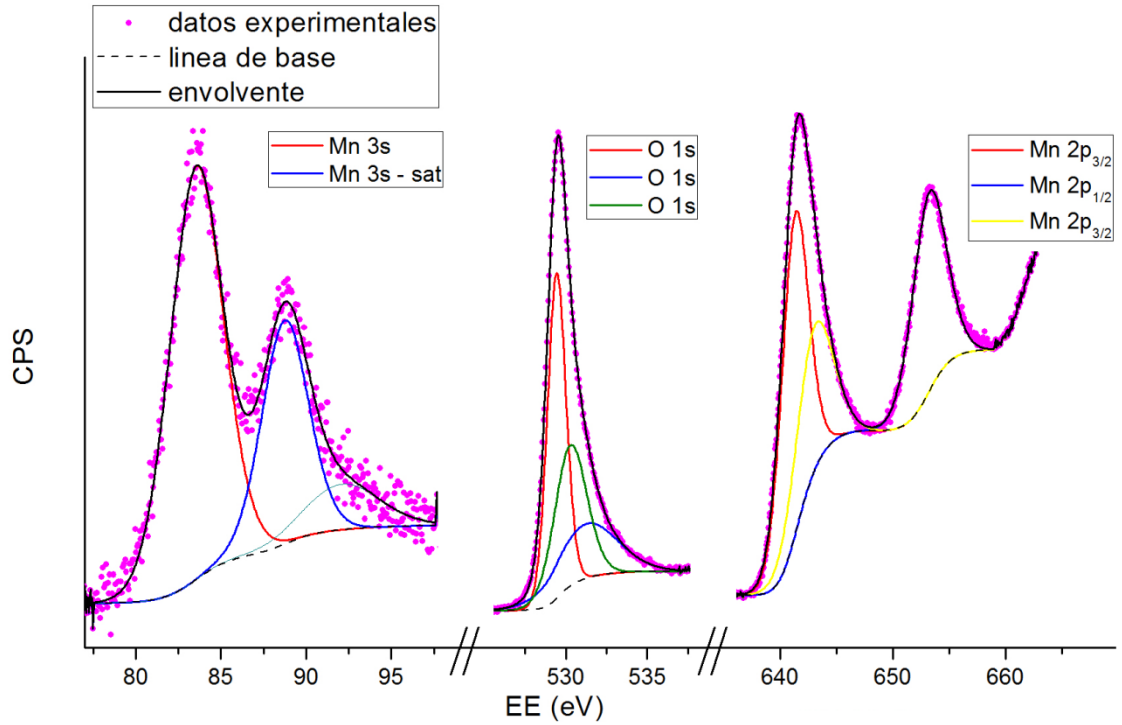


Figura 4: Curva de calibración para el CO_2.

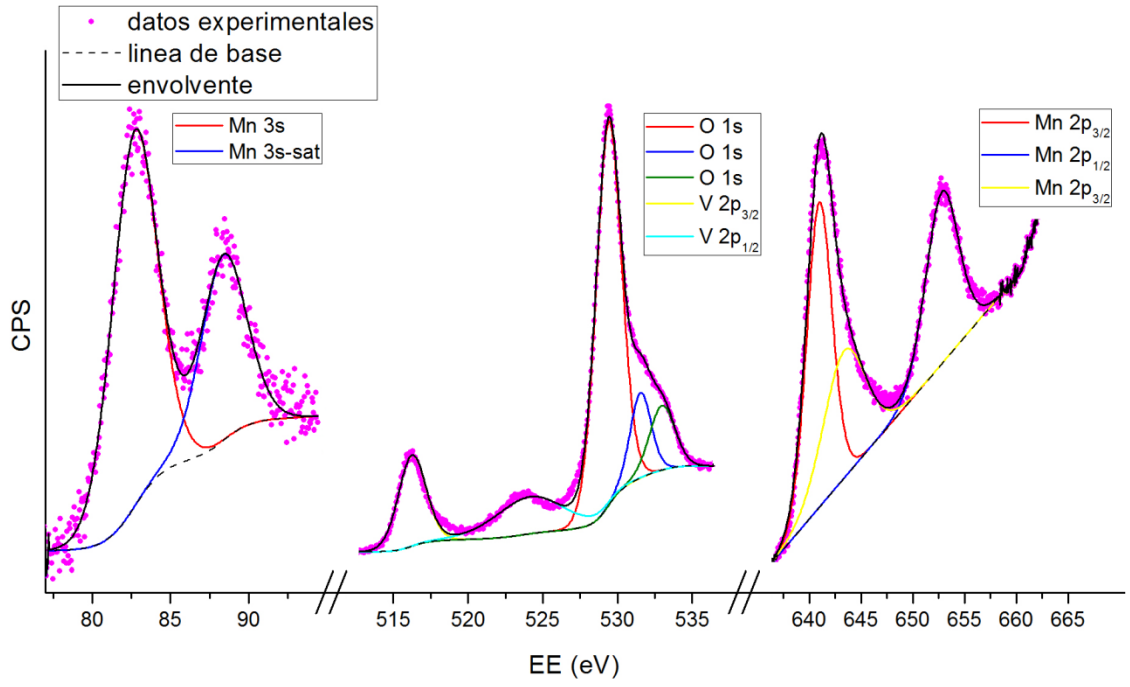
3. Apéndice III. Gráficos XPS

3.1 Catalizadores MnO_x(ac) y MnVY(ac)

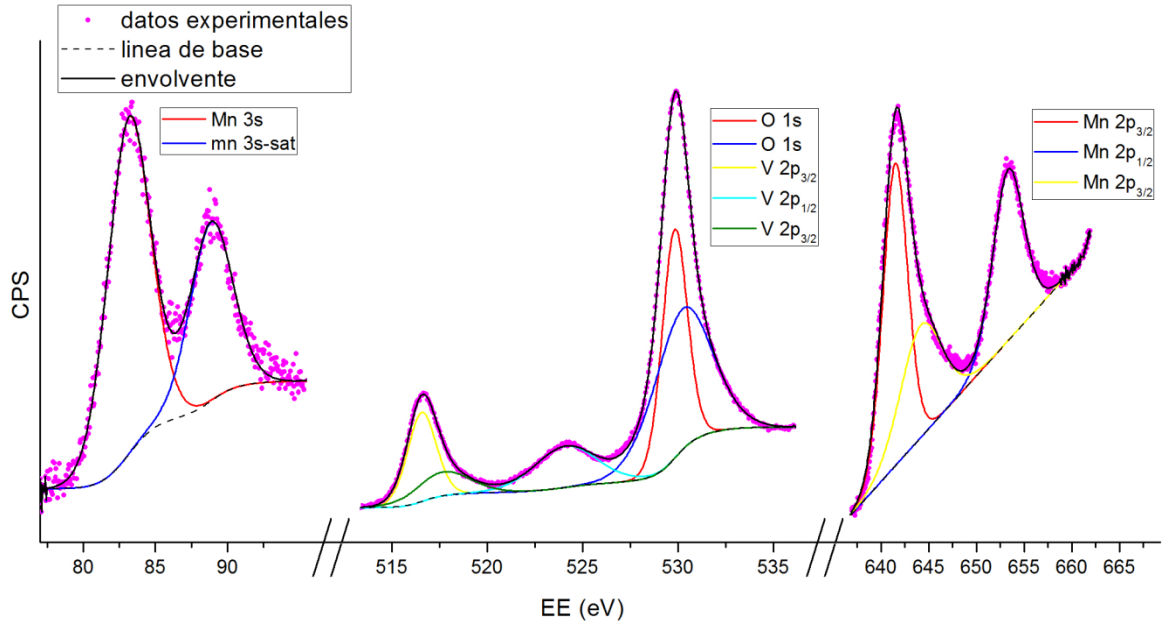
3.1.1 MnO_x(ac)



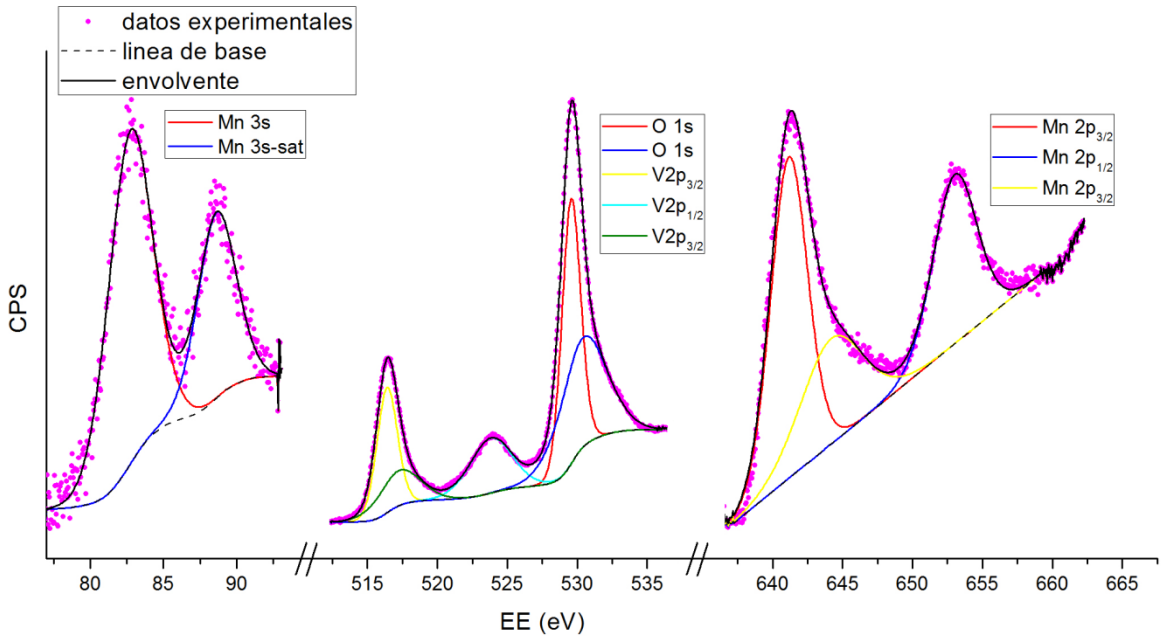
3.1.2 MnV12(ac)



3.1.3 MnV20(ac)

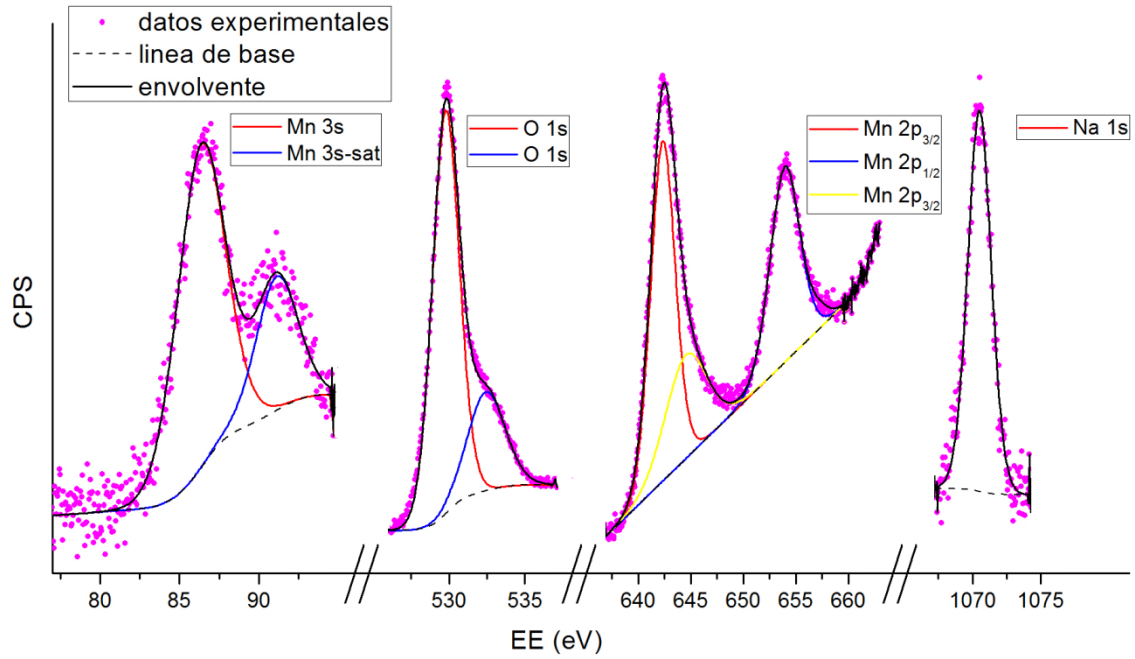


3.1.3 MnV30(ac)

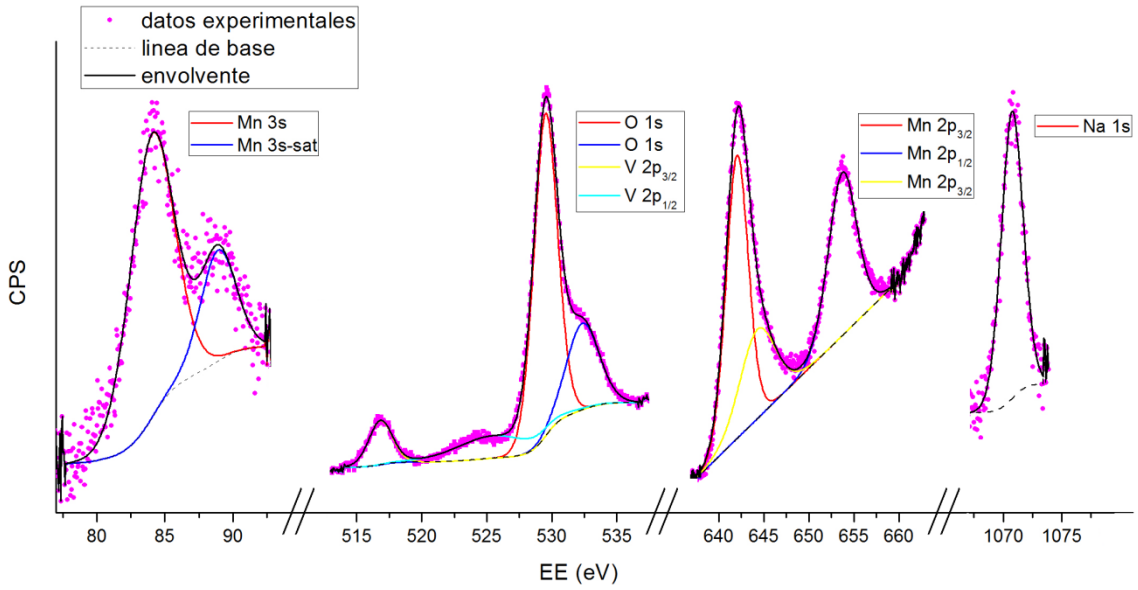


3.2 Catalizadores MnO_x(Na) y MnVY(Na)

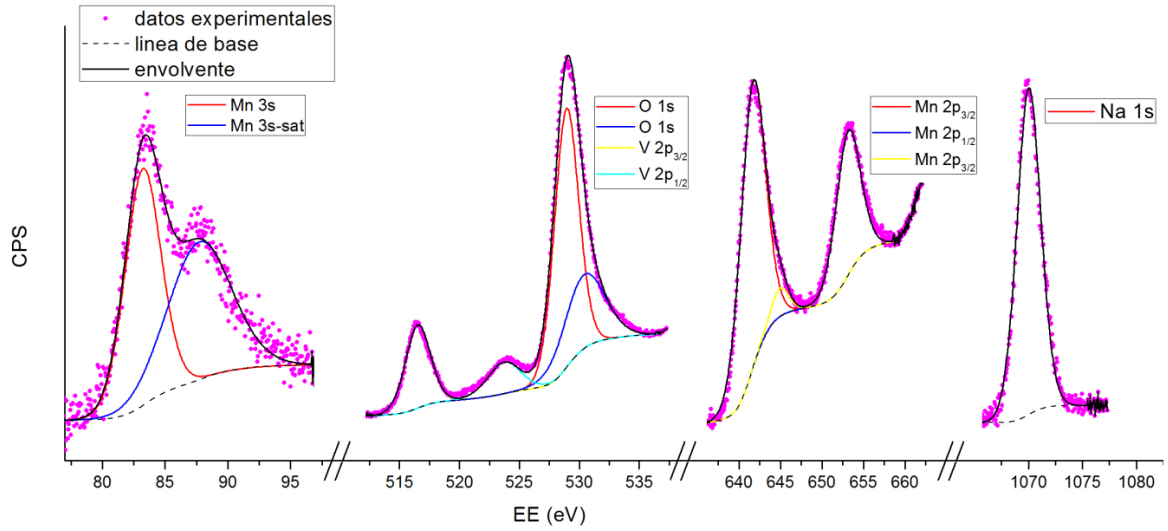
3.2.1 MnO_x(Na)



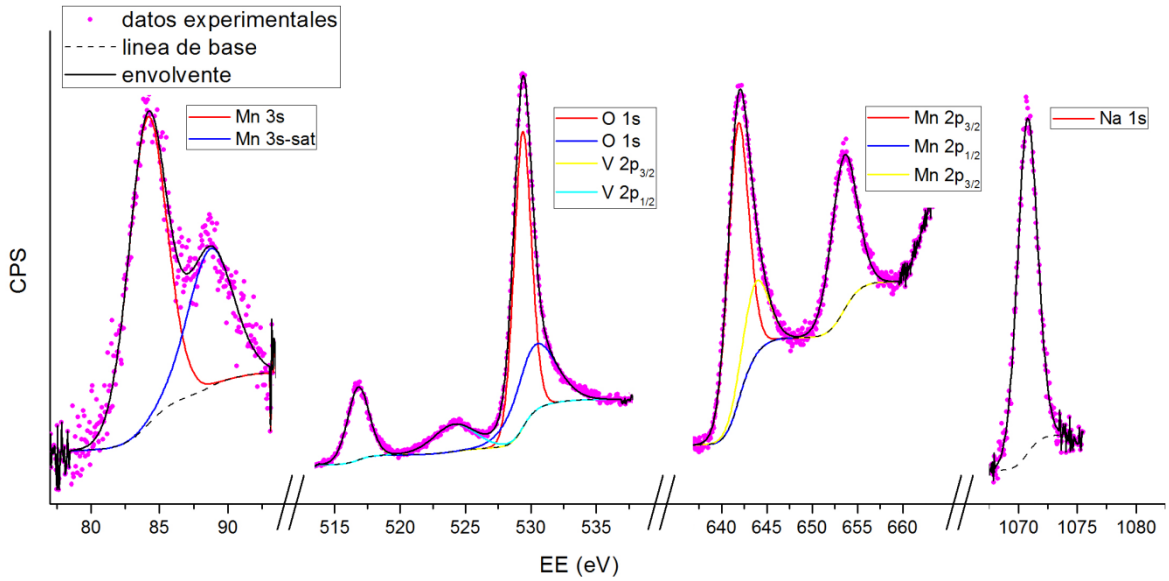
3.2.2 MnV12(Na)



3.2.3 MnV20(Na)

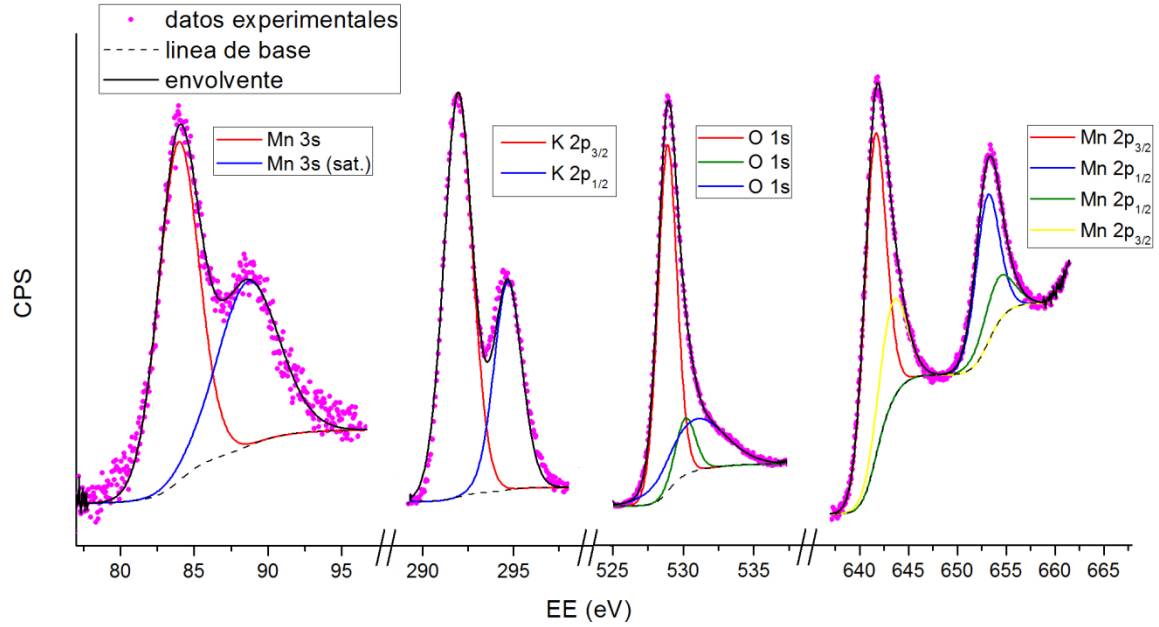


3.2.4 MnV30(Na)

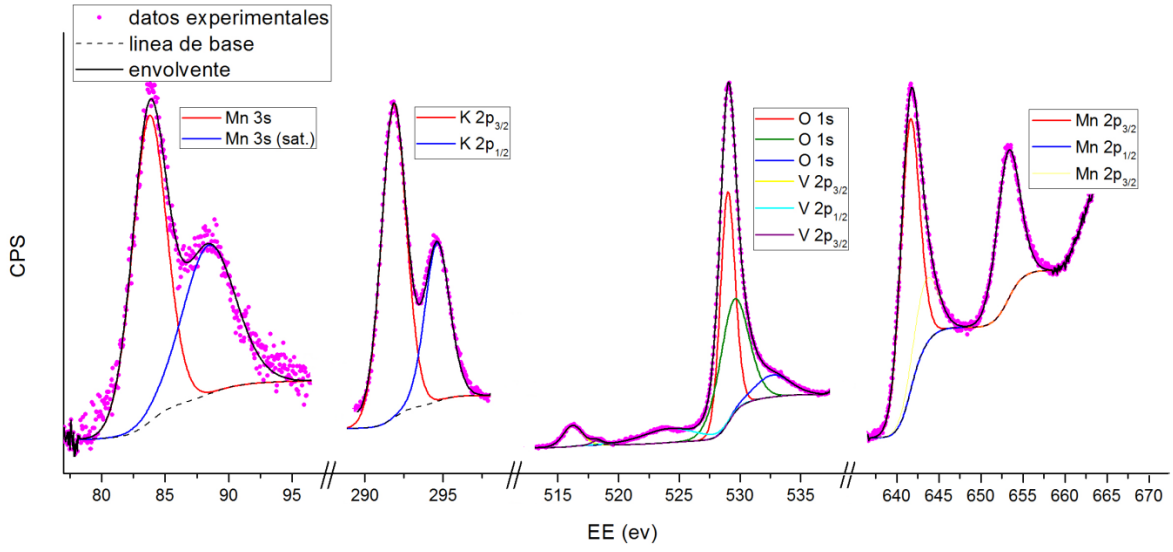


3.3 Catalizadores MnO_x(K) y MnVY(K)

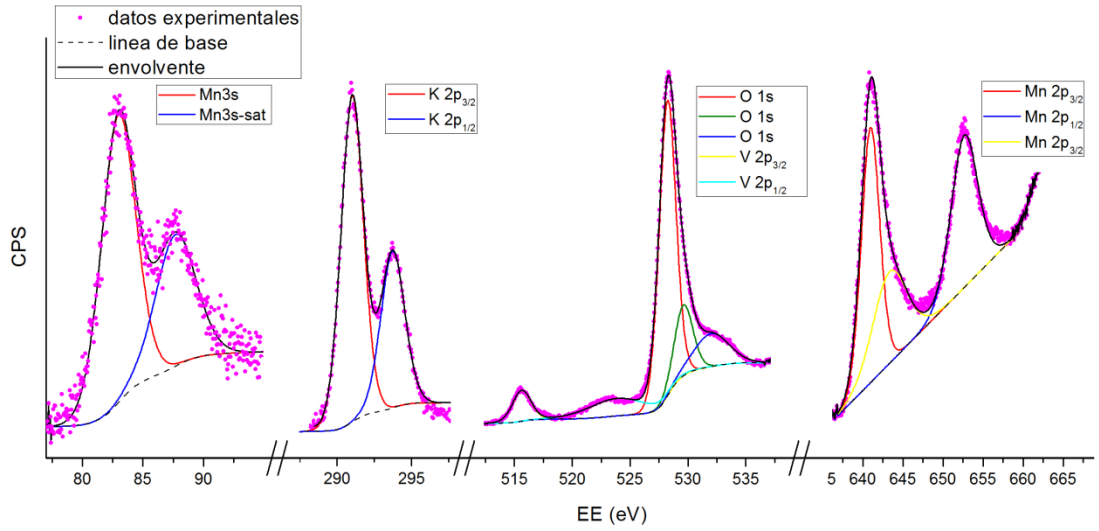
3.3.1 MnO_x(K)



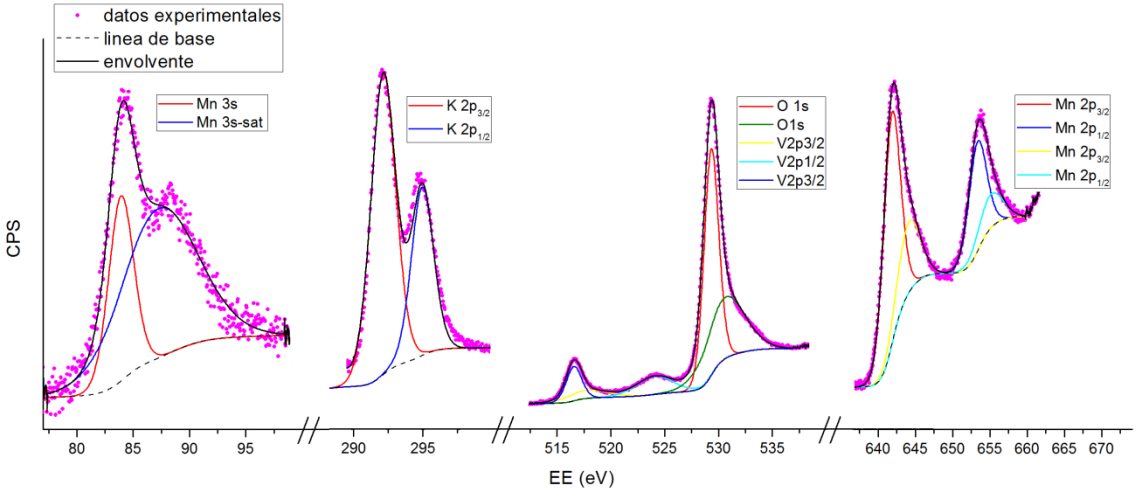
3.3.2 MnV12(K)



3.3.3 MnV20(K)

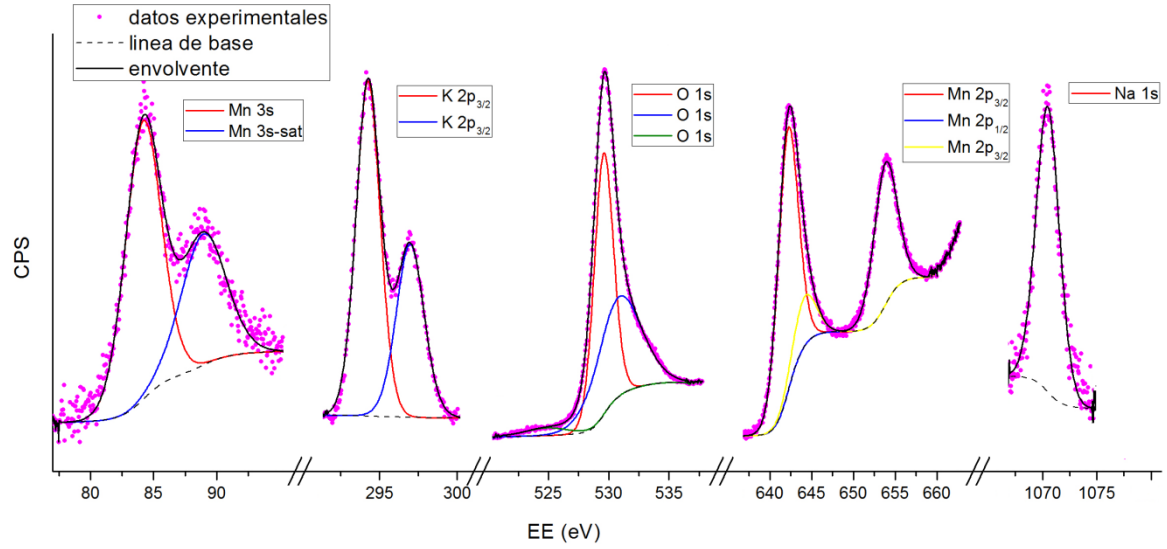


3.3.4 MnV30(K)

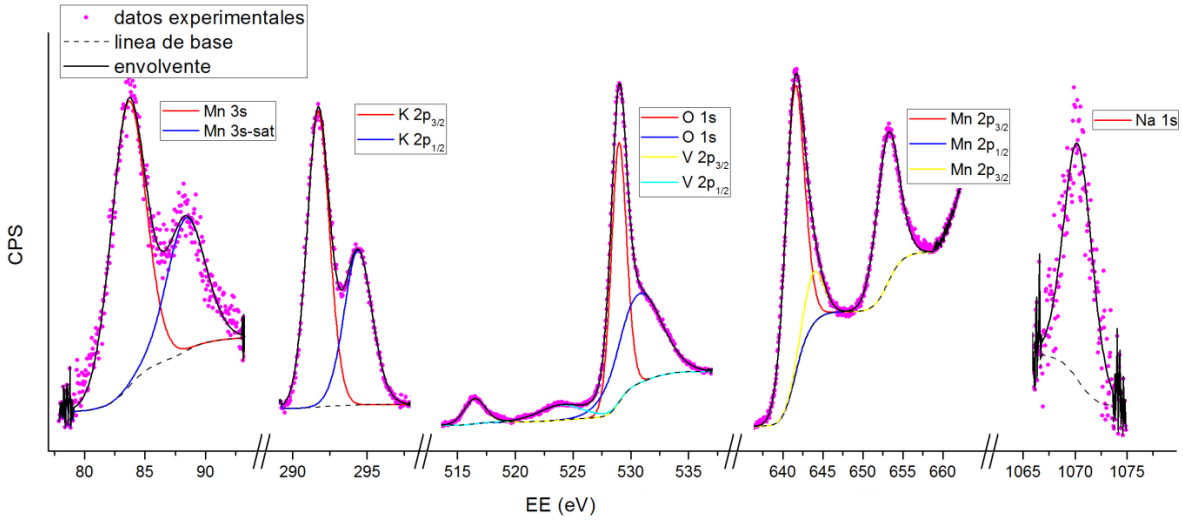


3.4 Catalizadores MnO_x(NaK) y MnVY(NaK)

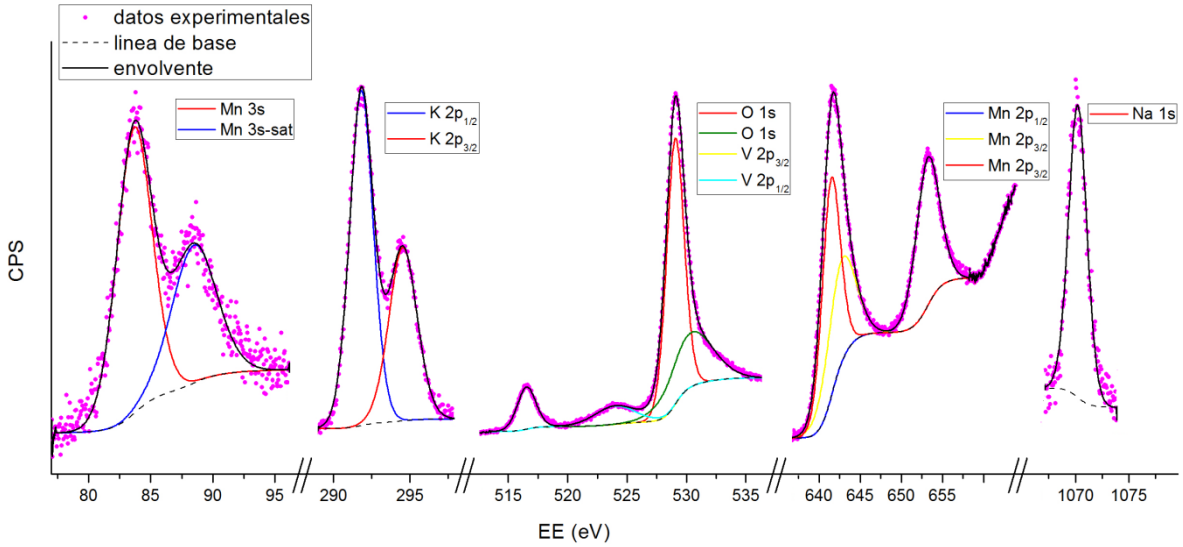
3.4.1 MnO_x(NaK)



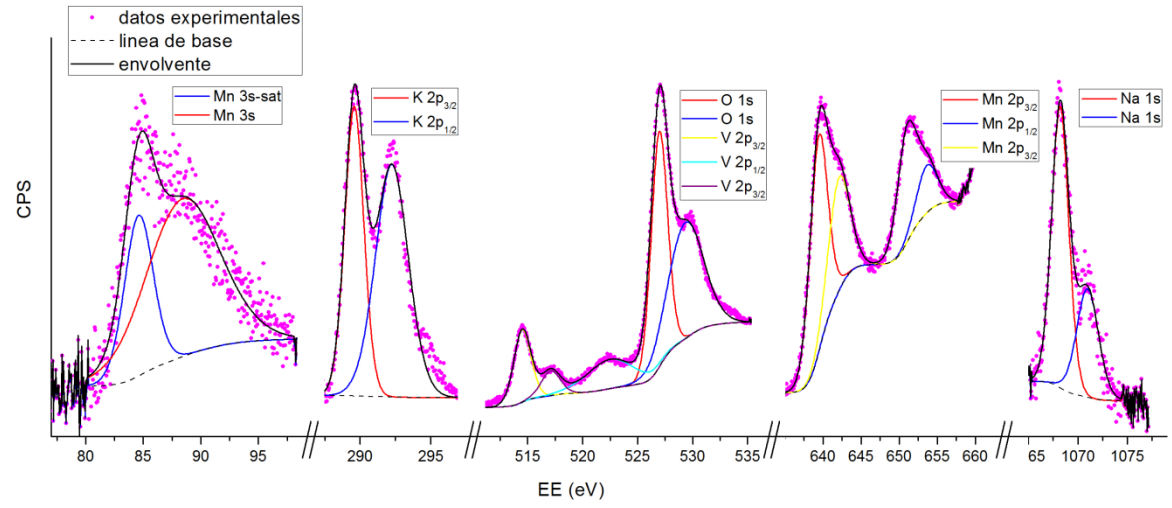
3.4.2 MnV12(NaK)



3.4.3 MnV20(NaK)



3.4.4 MnV30(NaK)



Apéndice IV. Tarjetas JCPDS

41-1442 Wavelength= 1.54178 *

Mn2O3

Manganese Oxide

Bixbyite-C, syn

Rad.: CuKa λ: 1.54178 Filter: Graph Mono d-sp: Diff.

Cut off: 15.0 Int.: Diffract. I/Icor.: 4.5

Ref: Syvinski, W., McCarthy, G., North Dakota State Univ., Fargo, ND, USA, ICDD Grant-in-Aid, (1990)

Sys.: Cubic S.G.: $Ia\bar{3}$ (206)

a: 9.4091(4) b: c: A: C:

α: β: γ: Z: 16 mp:

Ref: Geller, S., Acta Crystallogr., Sec. B, 27, 821 (1971)

Dx: 5.035 Dm: 4.950 SS/FOM: F_{30} = 118(.0082 , 31)

Color: Black
Peak height intensity. Sample prepared by heating Mn C O3 at 650 C for 48 hours. $\sigma(I_{obs})$= ±0.04. Isostructural with avicennite. Mn2 O3 type. Silicon used as an internal stand. PSC: cI80. To replace 2-896, 8-10, 10-69 and 31-825 and validated by calculated pattern. See ICSD 31112 (PDF 75-1573); ICSD 9091 (PDF 71-636). Mwt: 157.87. Volume[CD]: 833.00.

2θ	Int	h	k	l	2θ	Int	h	k	l
18.860	1	2	0	0	85.015	1	8	2	0
23.150	16	2	1	1	86.537	1	6	5	3
32.978	100	2	2	2	88.094	<1	8	2	2
35.714	2	3	2	1	89.624	1	8	3	1
38.266	14	4	0	0	91.174	2	6	6	2
40.650	1	4	1	1	92.700	<1	7	5	2
42.984	1	4	2	0	94.241	1	8	4	0
45.216	8	3	3	2	95.784	<1	8	3	3
47.339	1	4	2	2	97.340	<1	8	4	2
49.389	9	4	3	1	98.879	1	9	2	1
53.316	2	5	2	1	100.454	<1	6	6	4
55.237	28	4	4	0	102.007	<1	8	5	1
57.077	1	4	3	3	105.198	1	9	3	2
58.889	<1	6	0	0	106.805	1	8	4	4
60.672	3	6	1	1	108.395	1	9	4	1
62.425	1	6	2	0	110.032	<1	10	0	0
64.135	4	5	4	1	111.684	<1	10	1	1
65.864	13	6	2	2	113.349	1	10	2	0
67.520	3	6	3	1	115.027	<1	9	4	3
69.169	2	4	4	4	116.736	1	10	2	2
70.804	<1	5	4	3	118.479	1	10	3	1
72.416	1	6	4	0					
74.016	1	7	2	1					
75.618	1	6	4	2					
80.354	1	6	5	1					
81.910	1	8	0	0					
83.456	1	8	1	1					

80-0382 Wavelength= 1.54060 C

Mn3O4

Manganese Oxide

Hausmannite

Rad.: CuKa1 λ: 1.54060 Filter: d-sp: Calculated

Cut off: 17.7 Int.: Calculated I/Icor.: 2.79

Ref: Calculated from ICSD using POWD-12++, (1997)

Ref: Jarosch, D., Mineralogy and Petrology, 37, 15 (1987)

Sys.: Tetragonal S.G.: $I4_1/amd$ (141)

a: 5.765(1) b: c: 9.442(2) A: C: 1.6378

α: β: γ: Z: 4 mp:

Ref: Ibid.

Dx: 4.843 Dm:

Peak height intensity. Specimen from Langban, Sweden. R-factor: 0.034. Mn3 O4 type. PSC: tI28. See PDF 75-1560. Mwt: 228.81. Volume[CD]: 313.81.

2θ	Int	h	k	l	2θ	Int	h	k	l
18.014	277	1	0	1	72.300	14	3	3	2
28.915	382	1	1	2	73.390	12	4	2	0
30.999	161	2	0	0	74.146	83	4	1	3
32.383	764	1	0	3	76.549	39	4	2	2
36.084	999*	2	1	1	77.543	40	4	0	4
36.493	148	2	0	2	78.286	10	3	2	5
38.092	183	0	0	4	80.258	45	2	1	7
44.410	207	2	2	0	80.577	28	3	1	6
45.439	3	2	1	3	81.485	35	0	0	8
48.293	1	3	0	1	84.605	5	5	0	1
49.895	58	2	0	4	85.820	18	4	2	4
50.838	220	1	0	5	86.549	57	4	1	5
53.865	90	3	1	2	88.488	1	3	0	7
56.025	75	3	0	3	88.947	15	5	1	2
58.500	277	3	2	1	89.699	7	2	0	8
59.910	492	2	2	4					
60.746	9	2	1	5					
63.296	23	1	1	6					
64.616	178	4	0	0					
65.417	11	3	2	3					
67.793	26	2	0	6					
67.948	14	4	0	2					
69.766	45	3	0	5					
71.818	1	1	0	7					

71-0454 Wavelength= 1.54060 C

V307

Vanadium Oxide

Rad.: CuKa1 λ: 1.54060 Filter: d-sp: Calculated

Cut off: 17.7 Int.: Calculated I/Icor.: 1.67

Ref: Calculated from ICSD using POWD-12++, (1997)

Ref: Waltersson, K et al., Acta Crystallogr., Sec. B, 30, 2644 (1974)

Sys.: Monoclinic S.G.: C2/c (15)

a: 21.921(3) b: 3.679(1) c: 18.341(2) A: 5.9584 C: 4.9853

α: β: 95.610(1) γ: Z: 12 mp:

Ref: Ibid.

Dx: 3.585 Dm: 3.610

Peak height intensity. R-factor: 0.020. 07 V3 type. PSC: mC120. See PDF 27-940. Mwt: 264.82. Volume[CD]: 1472.07.

2θ	Int	h	k	l	2θ	Int	h	k	l	2θ	Int	h	k	l	2θ	Int	h	k	l
8.099	177	2	0	0	29.335	276	3	1	2	36.671	66	-8	0	4	49.512		0	2	0
9.683	1	0	0	2	29.335		0	0	6	37.753	55	-7	1	1	49.645	169	3	1	8
12.010	22	-2	0	2	29.681	186	-2	0	6	37.753		7	1	0	49.645		-9	1	5
13.233	1	2	0	2	29.862	51	-6	0	4	38.270	10	-1	1	6	49.797	48	-2	0	10
16.239	70	-4	0	0	30.337	115	-3	1	3	38.367	13	-7	1	2	49.797	48	-5	1	8
18.099	6	-4	0	2	31.261	112	-1	1	4	38.510	13	7	1	1	49.923	146	-7	1	7
19.437	88	0	0	4	31.261		2	0	6	38.616	7	5	1	4	49.923		0	0	10
19.747	251	4	0	2	31.507	104	3	1	3	38.912	1	-1	1	6	50.158	97	-12	0	0
20.331	7	-2	0	4	31.860	47	-5	1	1	39.149	7	-5	1	5	50.158		-12	0	2
21.817	282	2	0	4	32.236	31	-4	0	6	39.462	5	-3	1	6	50.259	52	2	2	0
24.156	45	-4	0	4	32.487	25	5	1	1	39.462		0	0	8	50.611	6	0	2	2
24.462	768	6	0	0	32.816	70	-8	0	0	39.617	20	-7	1	3	50.611		2	2	1
24.462		1	1	0	32.938	131	-3	1	4	39.849	3	7	1	2	50.773	3	9	1	4
24.926	999*	-1	1	1	32.938		6	0	4	40.112	40	8	0	4	51.154	52	-4	0	10
25.447	228	-6	0	2	34.242	40	-5	1	3	40.445	2	-6	0	6	51.154	52	-1	1	9
26.266	8	-1	1	2	34.320	25	-3	1	4	41.353	79	-4	0	8	51.524	7	2	2	2
26.567	3	1	1	2	34.544	49	-1	1	5	41.353	79	-10	0	0	51.524		2	0	10
27.142	3	3	1	0	35.128	176	4	0	6	41.456	45	-7	1	4	51.970	7	1	1	9
27.253	2	6	0	2	35.128		1	1	5	41.768	10	5	1	5	52.214	44	-12	0	2
27.369	17	-3	1	1	35.229	114	-8	0	2	41.768		7	1	3	52.214	44	-12	0	4
27.801	1	-3	1	1	35.946	41	-3	1	5	42.255	18	-8	0	6	52.356	32	-2	2	3
28.415	27	-1	1	3	35.946		-5	1	3	42.322	18	-1	1	7	52.356		11	1	0
28.415		-3	1	2	36.584	104	-6	0	6	42.322		-5	1	6	52.879	22	2	2	3
43.011	24	1	1	7	52.879		4	2	1	43.316	28	-3	1	7	53.222	4	5	1	8
43.552	41	10	0	2	53.481	16	-9	1	5	43.822	6	-7	1	5	53.481		-7	1	8
44.206	5	-10	0	4	53.680	12	0	2	4	44.206		7	1	4	53.809	35	4	2	2
44.480	4	-4	0	8	54.006	41	-5	1	9	44.599	3	-9	1	1	54.006	41	10	0	6
44.745	18	-9	1	0	54.152	19	3	1	9	44.745		-6	0	8	54.506	3	4	0	10
45.229	4	5	1	6	54.714	20	2	2	4	45.449	15	-9	1	1	54.888	14	-8	0	8
45.905	4	-5	1	7	55.073	4	-9	1	7	46.050	14	-9	1	3	55.298	16	-10	0	8
46.825	17	8	0	6	55.977	121	6	2	0	47.101	24	-7	1	5	55.977	121	11	1	3
47.475	5	-3	1	8	56.221	47	-2	2	5	47.597	7	-9	1	4	56.221		12	0	4
47.905	1	10	0	4	56.312	58	-12	0	6	48.504	4	9	1	3	56.477	51	6	2	1
49.132	45	5	1	7	56.477		-6	2	2	49.132		-6	0	8	56.588	17	9	1	6
49.512	256	-8	0	8	57.107	2	4	2	4	57.429	3	-7	1	9	57.429	3	-6	2	3
57.639	19	-5	1	9	57.859	11	-4	2	5	57.937	8	-8	0	10	58.024	6	7	1	8
58.130	6	11	1	4	58.597	43	-0	2	6	58.597		-11	1	6	58.729	73	-6	0	10
58.729		-2	2	6	58.901	40	-6	2	4	58.901	40	3	1	10	59.250	10	14	0	0
59.423	6	4	2	5	59.729	19	-2	2	6	60.334	14	-4	2	6	60.334		-13	1	1
60.539	10	-13	1	2	60.587	14	-2	0	12	60.587	14	-8	2	1	60.755	31	-6	2	4
60.755	31	-6	2	5	60.971	101	-1	1	11	60.971		-8	2	2	61.210	69	-13	1	3
61.210	69	14	0	2	61.437	36	-3	1	11	61.630	8	-4	0	12	61.630	8	-7	1	10
61.775	12	-2	2	7	61.775		-1	1	11	61.859	13	-8	2	3	62.036	35	-9	1	9
62.036		-12	0	8	62.216	19	-8	2	2	62.410	7	-2	0	12	63.110	36	-10	0	10
63.110	36	-6	2	6	63.662	4	-11	1	6	63.662		-8	2	3	63.917	14	-3	1	11
63.917	14	-6	0	12	64.142	17	-13	1	3	64.283	7	-11	1	8	64.915	1	-8	2	5
65.082	3	0	2	8	65.082	3	-2	2	8	65.257	5	4	0	12	65.257		4	2	7
65.539	13	-8	2	4	65.775	7	-6	2	6	66.170	28	-7	1	11	66.170	28	-1	1	12
66.417	23	-4	2	8	66.417		-10	2	0	66.588	13	-3	1	12	66.588		-10	2	2
66.970	15	-11	1	7	66.970		-10	2	1										

75-1224 — Wavelength= 1.54056 — C

Mn2V2O7

Manganese Vanadium Oxide

Rad.: CuKa1 λ: 1.54060 Filter: d-sp: Calculated

Cut off: 17.7 Int.: Calculated I/Icor.: 2.81

Ref: Calculated from ICSD using POWD-12++, (1997)

Ref: Nord, A.G., Neues Jahrb. Mineral., Monatsh., 1984, 283 (1984)

Sys.: Monoclinic S.G.: C2/m (12)

a: 6.179(1) b: 8.719(1) c: 4.966(1) A: 0.7087 C: 0.5696

α: β: 103.600(1) γ: Z: 2 mp:

Ref: Ibid.

Dx: 4.135 Dm: ICSD # : 030687

Peak height intensity. R-factor: 0.058. PSC: mC22. See PDF 38-34. At least one TF implausible. Mwt: 323.75. Volume[CD]: 260.04.

2θ	Int	h	k	l	2θ	Int	h	k	l
17.919	171	1	1	0	47.217	119	$\bar{2}$	2	2
18.366	16	0	0	1	48.595	7	$\bar{1}$	3	2
20.354	56	0	2	0	51.783	2	2	4	0
23.102	16	$\bar{1}$	1	1	52.684	31	$\bar{2}$	4	1
27.548	999*	0	2	1	53.126	11	$\bar{3}$	1	2
28.200	421	1	1	1	54.009	35	2	0	2
29.727	6	2	0	0	54.242	175	1	3	2
31.133	403	$\bar{2}$	0	1	54.578	17	3	1	1
34.248	97	1	3	0	54.770	40	1	5	0
36.297	163	2	2	0	55.234	17	$\bar{3}$	3	1
37.226	31	0	0	2	55.708	37	3	3	0
37.351	50	$\bar{1}$	3	1	56.576	1	$\bar{1}$	1	3
37.483	25	$\bar{2}$	2	1	56.873	8	0	4	2
38.163	47	$\bar{1}$	1	2	56.963	10	$\bar{1}$	5	1
38.834	53	2	0	1	57.208	12	0	0	3
40.850	96	1	3	1	58.048	7	$\bar{2}$	4	1
41.388	44	0	4	0	58.317	35	$\bar{2}$	0	3
42.126	52	$\bar{2}$	0	2	58.317		2	2	2
42.792	10	0	2	2	59.558	54	1	5	1
44.232	11	2	2	1	60.022	2	$\bar{4}$	0	1
44.826	24	1	1	2	60.534	12	$\bar{2}$	4	2
45.628	52	0	4	1	61.370	5	0	2	3
45.964	131	$\bar{3}$	1	1	61.629	75	$\bar{3}$	3	2
46.505	14	3	1	0	61.731	65	4	0	0

44-0141 — Wavelength= 1.54056 — *

α-MnO2

Manganese Oxide

Rad.: CuKa1 λ: 1.54056 Filter: Graph Mono d-sp: Diff.

Cut off: Int.: Diffract. I/Icor.:

Ref: Liles, D., Rossouw, M., Thackeray, M., Div. Mater. Sci. & Tech., CSIR, Pretoria, South Africa, Private Communication, (1992)

Sys.: Tetragonal S.G.: I4/m (87)

a: 9.7847(5) b: c: 2.8630(2) A: C: 0.2926

α: β: γ: Z: 8 mp:

Ref: Ibid.

Dx: 4.213 Dm: SS/FOM: F_{30} = 46(.0140 , 47)

Color: Black

Prepared by acid treatment of Li2 Mn O3 (prepared from electrolytic Mn O2 and Li O H A H2 O at 800 C) with 1-5M H2 S O4 at ~90 C and dried overnight at 300 C. Chemical analysis showed a residual Li+ content of only 0.029 wt.% and a residual H content of 0.03 wt.% (equivalent to a residual H2 O content of 0.29 wt.%). Validated by Rietveld structure refinements using both X-ray and neutron diffraction data. Mn O2 type. Silicon used as an internal stand. PSC: tI24. Mwt: 86.94. Volume[CD]: 274.10.

2θ	Int	h	k	l	2θ	Int	h	k	l
12.784	37	1	1	0	91.462	9	6	0	2
18.107	53	2	0	0	101.470	2	7	1	2
25.711	5	2	2	0	102.905	1	6	4	2
28.841	82	3	1	0	104.362	2	8	5	1
36.696	24	4	0	0	111.406	6	2	1	3
37.522	100	2	1	1	118.135	2	6	6	2
39.010	9	3	3	0	120.913	5	4	1	3
41.225	5	4	2	0					
41.968	28	3	0	1					
46.037	2	3	2	1					
47.373	8	5	1	0					
49.864	50	4	1	1					
52.855	<1	4	4	0					
56.372	29	6	0	0					
56.927	10	4	3	1					
60.274	58	5	2	1					
65.108	15	0	0	2					
66.691	1	1	1	2					
67.648	1	7	1	0					
68.185	5	2	0	2					
69.711	35	5	4	1					
71.189	<1	2	2	2					
72.711	8	3	1	2					
73.700	3	7	3	0					
77.160	4	4	0	2					
78.588	10	3	3	2					
83.818	2	6	6	0					
84.348	3	5	1	2					
87.148	2	7	4	1					

27-0749 Wavelength= 1.54056

Na2Mn5O10

Sodium Manganese Oxide

Rad.: λ: Filter: d-sp:

Cut off: Int.: Estimation I/Icor.:

Ref: Parant et al., J. Solid State Chem., 3, 1 (1971)

Sys.: Monoclinic S.G.:

a: 13.81 b: 2.863 c: 9.74 A: 4.8236 C: 3.4020

α: β: 95.3 γ: Z: 2 mp:

Ref: Ibid.

Dx: 4.163 Dm: 4.250 SS/FOM: F_{20} = 5(0.076 , 48)

Mwt: 480.66. Volume[CD]: 383.45.

2θ	Int	h	k	l
8.836	10	0	0	1
12.857	<5	2	0	0
16.463	100	2	0	1
18.239	20	0	0	2
18.865	10	$\bar{1}$	0	2
20.639	<5	$\bar{3}$	0	1
21.393	<5	$\bar{2}$	0	2
22.262	5	3	0	1
25.879	10	4	0	0
26.506	<5	$\bar{4}$	0	1
27.681	<5	$\bar{1}$	0	3
28.126	<5	3	0	2
30.441	20	$\bar{4}$	0	2
31.215	20	0	1	0
31.831	<5	1	1	0
33.114	<5	$\bar{1}$	1	1
35.093	5	$\bar{2}$	1	1
35.743	10	$\bar{5}$	0	2
36.403	<5	0	1	2
37.344	80	1	1	2

78-2265 Wavelength= 1.54060 C

Na(VO3)

Sodium Vanadium Oxide

Rad.: CuKa1 λ: 1.54060 Filter: d-sp: Calculated

Cut off: 17.7 Int.: Calculated I/Icor.: 1.67

Ref: Calculated from ICSD using POWD-12++, (1997)

Ref: Shaikh, A.M., Ferroelectrics, 107, 219 (1990)

Sys.: Monoclinic S.G.: Cc (9)

a: 10.557(2) b: 9.469(3) c: 5.88(1) A: 1.1149 C: 0.6210

α: β: 108.430(2) γ: Z: 8 mp:

Ref: Ibid.

Dx: 2.905 Dm:

Peak height intensity. R-factor: 0.035. C.D. Cell: a=10.333, b=9.469, c=5.880, β=104.24, a/b=1.0913, c/b=0.6210, S.G.=Ia(9). PSC: mC40. Mwt: 121.93. Volume[CD]: 557.64.

2θ	Int	h	k	l	2θ	Int	h	k	l
12.856	48	1	1	0	39.344	23	$\bar{4}$	2	1
17.697	346	2	0	0	40.039	95	$\bar{4}$	0	2
18.153	6	$\bar{1}$	1	1	40.732	1	4	2	0
18.727	172	0	2	0	41.401	135	0	4	1
22.576	34	1	1	1	41.981	10	$\bar{1}$	3	2
24.643	606	0	2	1	42.191	20	$\bar{2}$	4	0
25.877	160	$\bar{2}$	2	0	43.144	71	$\bar{2}$	4	1
27.320	999*	$\bar{2}$	2	1	43.944	35	$\bar{5}$	1	1
28.061	198	$\bar{3}$	1	1	44.547	15	$\bar{4}$	2	2
28.323	399	3	1	0	45.377	3	$\bar{3}$	3	2
29.652	4	1	3	0	45.895	5	3	3	1
31.366	134	$\bar{2}$	0	2	46.094	38	2	2	2
32.064	422	0	0	2	46.290	75	5	1	0
32.398	91	$\bar{1}$	3	1	46.398	52	1	3	2
33.411	293	2	2	1	47.475	37	$\bar{2}$	4	1
35.166	4	1	3	1	47.719	22	$\bar{5}$	1	2
35.834	1	4	0	0	47.719		$\bar{1}$	1	3
36.067	3	$\bar{3}$	1	2	48.247	4	4	2	1
36.691	98	3	1	1	48.907	35	$\bar{3}$	1	3
36.782	59	$\bar{2}$	2	2	48.907		1	5	0
37.294	28	1	1	2	49.672	40	3	1	2
37.390	18	0	2	2	50.052	40	$\bar{2}$	4	2
37.979	11	0	4	0	50.424	174	$\bar{2}$	2	3
39.053	25	$\bar{3}$	3	1	50.529	138	0	4	2
39.249	35	3	3	0	52.111	111	$\bar{4}$	4	1

2θ	Int	h	k	l	2θ	Int	h	k	l	2θ	Int	h	k	l	2θ	Int	h	k	l
52.111		$\bar{5}$	3	1	65.979	22	$\bar{7}$	1	0	76.610	6	$\bar{5}$	5	3	87.896	17	$\bar{1}$	7	3
52.720	11	1	5	1	66.162	39	$\bar{6}$	4	1	77.669	1	$\bar{1}$	7	2	88.178	4	$\bar{5}$	5	4
52.855	25	0	2	3	66.162		5	1	2	77.669		$\bar{2}$	6	3	88.382	2	9	1	0
53.206	23	4	4	0	66.274	17	$\bar{5}$	5	1	78.249	3	$\bar{4}$	4	4	88.382		$\bar{7}$	5	3
53.742	6	1	1	3	66.609	9	$\bar{2}$	2	4	79.053	1	8	2	0	88.665	4	2	4	4
54.146	16	5	1	1	67.056	21	0	0	4	79.506	2	2	2	4	88.665	4	$\bar{3}$	3	5
54.146		$\bar{5}$	3	0	67.829	38	$\bar{2}$	6	2	79.613	3	0	6	3	89.320	5	$\bar{3}$	7	2
54.358	15	$\bar{6}$	0	2	68.078	27	5	5	0	79.743	5	0	4	4	89.320		$\bar{9}$	3	2
54.850	9	$\bar{4}$	2	3	68.224	64	0	6	2	80.040	16	$\bar{3}$	7	2	89.694	12	$\bar{9}$	3	1
54.963	23	6	0	0	68.224		$\bar{6}$	4	2	80.040		$\bar{7}$	1	4	89.978	7	0	8	2
55.520	34	$\bar{5}$	3	2	68.756	3	$\bar{4}$	2	4	80.268	22	$\bar{7}$	3	1					
55.520		$\bar{1}$	3	3	68.756		6	4	0	80.268		$\bar{8}$	2	3					
55.612	26	$\bar{3}$	5	1	69.080	25	$\bar{7}$	3	1	80.412	5	3	7	1					
55.612		$\bar{6}$	2	1	69.246	15	$\bar{1}$	5	3	80.780	2	1	7	2					
55.762	29	3	5	0	69.547	9	$\bar{4}$	6	1	81.247	1	0	8	0					
56.369	4	$\bar{4}$	4	2	69.547		$\bar{5}$	1	4	81.247		$\bar{4}$	6	3					
56.583	14	4	0	2	69.764	13	$\bar{7}$	1	3	81.585	2	$\bar{5}$	1	3					
56.685	18	$\bar{5}$	1	3	70.105	2	$\bar{3}$	5	3	81.585		$\bar{7}$	5	1					
57.229	2	3	3	2	70.105		1	7	0	81.948	1	$\bar{6}$	6	1					
57.680	7	2	4	2	70.313	7	0	2	4	82.825	20	$\bar{7}$	5	2					
57.886	5	$\bar{1}$	5	2	70.313		$\bar{7}$	3	2	83.240	8	$\bar{9}$	1	2					
58.009	15	$\bar{6}$	2	2	70.474	8	4	6	0	83.408	10	0	8	1					
58.431	43	0	6	0	70.761	37	3	5	2	83.408		$\bar{3}$	5	4					
58.590	23	6	2	0	71.010	20	$\bar{3}$	3	4	83.744	16	3	1	4					
59.530	2	4	4	1	71.511	4	2	4	3	83.901	7	$\bar{6}$	6	2					
60.146	12	$\bar{4}$	2	2	71.922	12	1	1	4	83.901		$\bar{2}$	8	0					
60.615	35	$\bar{3}$	5	2	71.922		3	3	3	84.213	4	$\bar{1}$	5	4					
60.962	19	1	3	3	72.471	2	$\bar{7}$	3	0	84.588	21	$\bar{2}$	8	1					
60.962		0	6	1	72.646	1	$\bar{5}$	3	2	84.588	21	$\bar{8}$	4	2					
61.326	116	$\bar{5}$	3	1	73.194	7	$\bar{4}$	6	2	84.870	36	$\bar{7}$	5	0					
61.427	75	$\bar{2}$	4	3	73.194		1	7	1	84.870		$\bar{1}$	1	5					
61.427		1	5	2	73.366	7	$\bar{6}$	0	4	85.140	26	$\bar{5}$	7	1					
61.580	56	2	6	0	74.111	4	7	1	1	85.374	21	$\bar{2}$	2	5					
61.580		2	2	3	74.111		1	5	3	85.815	6	$\bar{5}$	1	5					
62.295	3	$\bar{2}$	6	1	74.340	3	2	6	2	85.815	6	$\bar{4}$	2	5					
62.422	10	$\bar{7}$	1	1	74.451	2	5	5	1	86.186	12	$\bar{7}$	3	4					
63.259	25	$\bar{2}$	0	4	74.977	4	$\bar{8}$	2	1	86.186		$\bar{7}$	1	2					
63.576	22	0	4	3	75.389	1	$\bar{8}$	2	2	86.419	6	$\bar{8}$	0	4					
63.733	75	$\bar{5}$	3	3	75.692	2	$\bar{3}$	7	1	86.807	5	5	7	0					
63.733		$\bar{7}$	1	2	75.819	2	3	7	0	87.255	2	$\bar{6}$	4	2					
64.451	16	$\bar{3}$	1	4	75.945	2	8	0	0	87.255		$\bar{9}$	1	3					
65.185	3	$\bar{6}$	2	3	75.945	2	4	6	1	87.459	5	8	2	1					
65.454	34	$\bar{3}$	1	3	76.114	12	$\bar{7}$	3	3	87.682	32	2	8	1					
65.454		$\bar{4}$	0	4	76.404	6	2	0	4	87.682		5	3	3					

72-1982 Wavelength= 1.54060 C

α-MnO2

Manganese Oxide

Rad.: CuKa1 λ: 1.54060 Filter: d-sp: Calculated

Cut off: 17.7 Int.: Calculated I/Icor.: 3.52

Ref: Calculated from ICSD using POWD-12++, (1997)

Ref: Kondrashev, Y.D., Zaslavsky, A.I., Izv. Akad. Nauk SSSR, Ser. Fiz., 15, 179 (1951)

Sys.: Tetragonal S.G.: I4/m (87)

a: 9.815(1) b: c: 2.847(1) A: C: 0.2901

α: β: γ: Z: 8 mp:

Ref: Ibid.

Dx: 4.211 Dm:

Peak height intensity. Mn O2 type. PSC: tI24. See PDF 42-1348.
No R value given. At least one TF missing. Mwt: 86.94.
Volume[CD]: 274.26.

2θ	Int	h	k	l	2θ	Int	h	k	l
12.745	999*	1	1	0	68.935	17	6	4	0
18.061	772	2	0	0	69.603	114	4	5	1
25.651	24	2	2	0	71.590	1	2	2	2
28.740	378	3	1	0	72.588	2	6	3	1
32.726	1	1	0	1	73.072	26	3	1	2
36.592	138	4	0	0	73.411	18	3	7	0
37.628	421	1	2	1	75.524	1	7	0	1
38.898	71	3	3	0	77.452	15	4	0	2
41.095	22	4	2	0	78.422	16	7	2	1
42.036	105	3	0	1	78.894	13	3	3	2
46.097	5	2	3	1	80.330	3	2	4	2
47.179	48	5	1	0	83.509	9	6	6	0
49.897	202	4	1	1	84.138	8	5	6	1
52.714	3	4	4	0	84.602	8	5	1	2
54.468	5	5	3	0	84.928	11	5	7	0
56.185	88	6	0	0	86.969	20	1	8	1
56.933	43	5	0	1	88.846	1	4	4	2
59.519	25	2	6	0	89.170	2	4	8	0
60.242	177	5	2	1					
65.521	39	0	0	2					
66.560	8	1	6	1					
67.063	9	1	1	2					
67.415	17	7	1	0					
68.587	16	2	0	2					

34-0168 Wavelength= 1.5406 C

KMn8O16

Potassium Manganese Oxide

Cryptomelane-M

Rad.: CuKa1 λ: 1.5406 Filter: d-sp: Calculated

Cut off: 22.1 Int.: Calculated I/Icor.: 0.92

Ref: Smith, D., Zolensky, Penn State University, University Park, Pennsylvania, USA, ICDD Grant-in-Aid, (1983)

Sys.: Monoclinic S.G.: I2/m (12)

a: 9.956(3) b: 2.8705(9) c: 9.706(4) A: 3.4684 C: 3.3813

α: β: 90.95(3) γ: Z: 1 mp:

Ref: Post, J. et al., Acta Crystallogr., Sec. B, 38, 1056 (1982)

Dx: 4.582 Dm: SS/FOM: F_{30} = 364(.0023 , 36)

Integrated intensities. Specimen from Chindwara, India (NMNH 89104). Microprobe analysis (wt.%): Na2 O 1.02, Al2 O3 0.99, K2 O 5.78, Mn2 O3/Mn O2 84.41, Fe2 O3 3.03, SrO 1.75, BaO 1.97, minor MgO. Cryptomelane SuperGroup, 2M Group. PSC: mC25.42. Deleted by 44-1386, experimental confirms calculated, PB 2/93. Volume[CD]: 277.35.

2θ	Int	h	k	l	2θ	Int	h	k	l
12.623	79	$\bar{1}$	0	1	47.859	2	1	0	5
12.834	82	1	0	1	49.201	24	$\bar{4}$	1	1
17.807	72	2	0	0	49.449	22	4	1	1
18.270	91	0	0	2	49.909	19	$\bar{1}$	1	4
25.399	15	$\bar{2}$	0	2	50.153	32	1	1	4
25.833	15	2	0	2	52.172	3	$\bar{4}$	0	4
28.254	43	$\bar{3}$	0	1	53.112	3	4	0	4
28.550	99	3	0	1	54.316	3	$\bar{3}$	0	5
28.852	100	$\bar{1}$	0	3	54.473	3	5	0	3
29.141	44	1	0	3	55.328	24	6	0	0
32.497	3	0	1	1	56.178	3	$\bar{5}$	1	0
36.056	25	4	0	0	56.532	3	3	1	4
37.026	22	0	0	4	56.883	25	0	0	6
37.217	54	$\bar{2}$	1	1	57.199	15	3	1	4
37.377	99	2	1	1	57.250	4	0	1	5
37.555	77	1	1	2	58.442	3	$\bar{6}$	0	2
38.507	10	$\bar{3}$	0	3	59.317	14	$\bar{5}$	1	2
39.188	6	3	0	3	59.859	32	5	1	2
40.435	5	$\bar{4}$	0	2	60.190	28	$\bar{2}$	1	5
41.089	20	$\bar{2}$	0	4	60.424	17	2	0	6
41.564	36	3	1	0	60.726	15	2	1	5
42.051	33	0	1	3	64.916	19	0	2	0
45.554	5	$\bar{3}$	1	2	65.501	5	$\bar{6}$	1	1
46.194	4	2	1	3	66.565	4	7	0	1
46.366	7	$\bar{5}$	0	1	67.176	2	1	1	6
46.687	3	5	0	1	68.069	6	0	2	2
47.543	5	$\bar{1}$	0	5	68.711	25	$\bar{5}$	1	4

2θ	Int	h	k	l	2θ	Int	h	k	l
69.051		$\bar{4}$	1	5	100.103	2	$\bar{6}$	1	7
69.051	16	6	0	4	101.629	3	$\bar{7}$	1	6
69.707		5	1	4	102.070	4	$\bar{8}$	1	5
69.707	7	4	0	6	102.632	3	6	2	4
70.052	18	$\bar{4}$	1	5	102.952	2	7	0	7
72.039	5	$\bar{7}$	0	3	103.512	2	$\bar{5}$	1	8
72.256		$\bar{3}$	2	1	103.989	3	8	1	5
72.256	7	6	1	3	105.533	4	$\bar{7}$	2	3
72.404		3	2	1	107.025	2	$\bar{3}$	2	7
72.404	9	$\bar{3}$	1	6	108.057	2	3	2	7
72.558		$\bar{1}$	2	3	108.424	2	2	0	10
72.558	10	1	2	3	110.944	3	2	3	1
73.066	4	7	0	3	114.850	2	1	1	10
73.310	3	3	1	6	116.492	2	$\bar{6}$	2	6
73.542	3	$\bar{3}$	0	7	118.396	3	6	2	6
74.562	4	3	0	7	120.758	3	1	3	4
76.568	4	4	2	0	124.228	2	9	2	1
77.155	3	0	2	4	125.568	2	$\bar{7}$	0	9
77.700	2	7	1	2	126.373	2	11	1	0
78.093	3	$\bar{3}$	2	3	127.223	3	$\bar{1}$	2	9
78.521	3	3	2	3	127.819	3	$\bar{9}$	0	7
78.675	2	$\bar{2}$	1	7	128.814	2	$\bar{10}$	0	6
79.346		$\bar{4}$	2	2	129.378	2	$\bar{11}$	1	2
79.346	2	2	1	7	130.710	6	11	1	2
79.766		4	2	2	132.091	3	9	2	3
79.766	2	$\bar{2}$	2	4	132.615	3	10	1	5
80.143	2	2	2	4	133.084	3	$\bar{5}$	1	10
82.533	2	$\bar{6}$	0	6	133.911	3	$\bar{3}$	2	9
84.224		6	0	6	135.055	3	$\bar{2}$	1	11
84.224	4	$\bar{1}$	2	5	135.693		3	2	9
84.405	3	$\bar{5}$	0	7	135.693	3	6	0	10
85.397	3	$\bar{8}$	1	1	136.514	2	2	1	11
85.771	2	8	1	1	140.949		$\bar{11}$	1	4
87.661	2	$\bar{1}$	1	8	140.949	4	[$\bar{8}$	2	6]
88.029	3	1	1	8	142.117	4	$\bar{5}$	3	4
89.216	3	9	0	1					
89.589		$\bar{3}$	2	5					
89.589	2	5	2	3					
90.409	6	6	2	0					
90.633	4	4	0	8					
91.707	8	0	2	6					
91.927	6	8	1	3					

89-3857 Wavelength= 1.54060 C

K(VO3)

Potassium Vanadium Oxide

Rad.: CuKa1 λ: 1.54060 Filter: d-sp: Calculated

Cut off: 17.7 Int.: Calculated I/Icor.: 3.09

Ref: Calculated from ICSD using POWD-12++

Ref: Petrasova, M., Madar, J., Hanic, F., Chem. Zvesti, 12, 410 (1958)

Sys.: Orthorhombic S.G.: Pmab (57)

a: 5.7(1) b: 10.83(3) c: 5.19(1) A: 0.5263 C: 0.4792

α: β: γ: Z: 4 mp:

Ref: Ibid.

Dx: 2.862 Dm:

Peak height intensity. R-factor: 0.145. PSC: oP20. At least one TF missing. Mwt: 138.04. Volume[CD]: 320.38.

2θ	Int	h	k	l	2θ	Int	h	k	l
16.356	80	0	2	0	44.046	25	2	3	1
17.071	155	0	0	1	45.332	22	0	5	1
18.946	4	0	1	1	46.211	113	2	4	0
22.631	120	1	2	0	47.338	66	2	0	2
23.727	158	0	2	1	48.122	30	2	1	2
24.591	47	1	1	1	48.205	23	1	5	1
28.485	999*	1	2	1	48.556	26	0	4	2
30.131	5	0	3	1	49.613	1	2	4	1
31.362	382	2	0	0	50.417	2	2	2	2
33.059	66	0	4	0	50.524	6	0	6	0
34.070	142	1	3	1	50.891	8	3	2	0
34.536	145	0	0	2	51.292	58	1	4	2
35.568	10	0	1	2	51.896	4	3	1	1
35.568		2	2	0	52.880	6	0	0	3
35.920	1	2	0	1	53.186	8	1	6	0
36.718	144	1	4	0	53.722	39	0	6	1
37.434	45	0	4	1	54.074	110	3	2	1
38.436	15	0	2	2	54.074		2	3	2
39.002	107	1	1	2	55.193	8	0	5	2
39.703	2	2	2	1	55.735	12	0	2	3
40.753	6	1	4	1	56.162	24	2	5	1
41.688	6	1	2	2	56.162		1	1	3
42.886	21	0	3	2	56.277	17	1	6	1

2θ	Int	h	k	l	2θ	Int	h	k	l
57.578	19	3	3	1	76.152	24	4	0	2
57.704	17	1	5	2	76.152		0	6	3
58.231	57	1	2	3	76.451	22	3	2	3
58.950	41	2	4	2	76.748	10	4	1	2
59.174	23	0	3	3	77.403	3	1	2	4
59.377	26	3	4	0	77.898	4	4	4	1
60.682	19	2	6	0	78.227	8	0	3	4
60.993	17	3	1	2	78.227	8	1	6	3
61.582	28	1	3	3	78.529	5	4	2	2
62.269	2	3	4	1	79.415	11	3	3	3
62.602	5	0	7	1	79.521	10	2	7	2
62.602		0	6	2	80.358	2	1	3	4
62.782	10	2	0	3	80.660	9	2	8	1
62.963	6	3	2	2	81.472	4	2	0	4
63.539	72	2	6	1	81.472		4	3	2
63.794	39	0	4	3	82.018	25	1	8	2
64.976	3	1	7	1	82.018		2	1	4
64.976		1	6	2	82.161	13	0	9	1
65.444	42	2	2	3	82.484	2	3	6	2
65.444		4	0	0	83.189	3	4	5	1
66.104	17	1	4	3	83.520	7	3	4	3
66.104		3	3	2	83.817	12	2	2	4
68.021	4	4	2	0	83.817		0	7	3
68.021		3	5	1	84.452	4	1	4	4
68.190	4	4	0	1	84.656	12	2	6	3
68.511	19	2	3	3	85.557	6	4	4	2
69.480	5	0	5	3	85.918	3	1	7	3
70.540	17	3	4	2	86.679	3	2	3	4
70.667	10	4	2	1	87.057	1	4	6	0
70.853	6	0	7	2	87.341	1	5	2	0
71.584	36	1	8	0	87.585	4	0	5	4
71.699	23	1	5	3	88.127	1	5	1	1
71.699	23	2	6	2	88.641	16	3	8	0
72.120	3	3	6	0	88.907	10	4	0	3
72.812	5	2	4	3	89.582	13	4	1	3
72.812		0	0	4	89.582		4	6	1
73.054	3	1	7	2	89.678	11	1	5	4
73.443	6	0	1	4	89.678		0	9	2
73.712	3	4	3	1	89.867	22	5	2	1
74.225	2	1	8	1					
74.655	7	3	1	3					
74.754	4	3	6	1					
75.303	7	0	2	4					
75.303		4	4	0					
75.615	4	1	1	4					
75.990	5	3	5	2					

18-1035 Wavelength= 1.5406

KMnO2

Potassium Manganese Oxide

Rad.: λ: Filter: d-sp:

Cut off: Int.: Estimation I/Icor.:

Ref: Lehmann, Teske, Z. Anorg. Allg. Chem., 336, 197 (1965)

Sys.: S.G.:

a: b: c: A: C:

α: β: γ: Z: mp:

Ref:

Dx: Dm: SS/FOM: F =

2θ	Int	h	k	l
12.581	100			
25.208	100			
29.258	10			
32.533	60			
35.023	10			
36.650	60			
40.227	30			
42.402	60			

Deleted by 44-1025; unindexed; Weissmann 5/93. Mwt: 126.04.

34-0168 Wavelength= 1.5406 C

KMn8O16

Potassium Manganese Oxide

Cryptomelane-M

Rad.: CuKa1 λ: 1.5406 Filter: d-sp: Calculated

Cut off: 22.1 Int.: Calculated I/Icor.: 0.92

Ref: Smith, D., Zolensky, Penn State University, University Park, Pennsylvania, USA, ICDD Grant-in-Aid, (1983)

Sys.: Monoclinic S.G.: I2/m (12)

a: 9.956(3) b: 2.8705(9) c: 9.706(4) A: 3.4684 C: 3.3813

α: β: 90.95(3) γ: Z: 1 mp:

Ref: Post, J. et al., Acta Crystallogr., Sec. B, 38, 1056 (1982)

Dx: 4.582 Dm: SS/FOM: F_{30} = 364(.0023 , 36)

Integrated intensities. Specimen from Chindwara, India (NMNH 89104). Microprobe analysis (wt.%): Na2 O 1.02, Al2 O3 0.99, K2 O 5.78, Mn2 O3/Mn O2 84.41, Fe2 O3 3.03, SrO 1.75, BaO 1.97, minor MgO. Cryptomelane SuperGroup, 2M Group. PSC: mC25.42. Deleted by 44-1386, experimental confirms calculated, PB 2/93. Volume[CD]: 277.35.

2θ	Int	h	k	l	2θ	Int	h	k	l
12.623	79	$\bar{1}$	0	1	47.859	2	1	0	5
12.834	82	1	0	1	49.201	24	$\bar{4}$	1	1
17.807	72	2	0	0	49.449	22	4	1	1
18.270	91	0	0	2	49.909	19	$\bar{1}$	1	4
25.399	15	$\bar{2}$	0	2	50.153	32	1	1	4
25.833	15	2	0	2	52.172	3	$\bar{4}$	0	4
28.254	43	$\bar{3}$	0	1	53.112	3	4	0	4
28.550	99	3	0	1	54.316	3	$\bar{3}$	0	5
28.852	100	$\bar{1}$	0	3	54.473	3	5	0	3
29.141	44	1	0	3	55.328	24	6	0	0
32.497	3	0	1	1	56.178	3	$\bar{5}$	1	0
36.056	25	4	0	0	56.532	3	3	1	4
37.026	22	0	0	4	56.883	25	0	0	6
37.217	54	$\bar{2}$	1	1	57.199	15	3	1	4
37.377	99	2	1	1	57.250	4	0	1	5
37.555	77	1	1	2	58.442	3	$\bar{6}$	0	2
38.507	10	$\bar{3}$	0	3	59.317	14	$\bar{5}$	1	2
39.188	6	3	0	3	59.859	32	5	1	2
40.435	5	$\bar{4}$	0	2	60.190	28	$\bar{2}$	1	5
41.089	20	$\bar{2}$	0	4	60.424	17	2	0	6
41.564	36	3	1	0	60.726	15	2	1	5
42.051	33	0	1	3	64.916	19	0	2	0
45.554	5	$\bar{3}$	1	2	65.501	5	$\bar{6}$	1	1
46.194	4	2	1	3	66.565	4	7	0	1
46.366	7	$\bar{5}$	0	1	67.176	2	1	1	6
46.687	3	5	0	1	68.069	6	0	2	2
47.543	5	$\bar{1}$	0	5	68.711	25	$\bar{5}$	1	4

2θ	Int	h	k	l	2θ	Int	h	k	l
69.051		$\bar{4}$	1	5	100.103	2	$\bar{6}$	1	7
69.051	16	6	0	4	101.629	3	$\bar{7}$	1	6
69.707		5	1	4	102.070	4	$\bar{8}$	1	5
69.707	7	4	0	6	102.632	3	6	2	4
70.052	18	$\bar{4}$	1	5	102.952	2	7	0	7
72.039	5	$\bar{7}$	0	3	103.512	2	$\bar{5}$	1	8
72.256		$\bar{3}$	2	1	103.989	3	8	1	5
72.256	7	6	1	3	105.533	4	$\bar{7}$	2	3
72.404		3	2	1	107.025	2	$\bar{3}$	2	7
72.404	9	$\bar{3}$	1	6	108.057	2	3	2	7
72.558		$\bar{1}$	2	3	108.424	2	2	0	10
72.558	10	1	2	3	110.944	3	2	3	1
73.066	4	7	0	3	114.850	2	1	1	10
73.310	3	3	1	6	116.492	2	$\bar{6}$	2	6
73.542	3	$\bar{3}$	0	7	118.396	3	6	2	6
74.562	4	3	0	7	120.758	3	1	3	4
76.568	4	4	2	0	124.228	2	9	2	1
77.155	3	0	2	4	125.568	2	$\bar{7}$	0	9
77.700	2	7	1	2	126.373	2	11	1	0
78.093	3	$\bar{3}$	2	3	127.223	3	$\bar{1}$	2	9
78.521	3	3	2	3	127.819	3	$\bar{9}$	0	7
78.675	2	$\bar{2}$	1	7	128.814	2	$\bar{10}$	0	6
79.346		$\bar{4}$	2	2	129.378	2	$\bar{11}$	1	2
79.346	2	2	1	7	130.710	6	11	1	2
79.766		4	2	2	132.091	3	9	2	3
79.766	2	$\bar{2}$	2	4	132.615	3	10	1	5
80.143	2	2	2	4	133.084	3	$\bar{5}$	1	10
82.533	2	$\bar{6}$	0	6	133.911	3	$\bar{3}$	2	9
84.224		6	0	6	135.055	3	$\bar{2}$	1	11
84.224	4	$\bar{1}$	2	5	135.693		3	2	9
84.405	3	$\bar{5}$	0	7	135.693	3	6	0	10
85.397	3	$\bar{8}$	1	1	136.514	2	2	1	11
85.771	2	8	1	1	140.949		$\bar{11}$	1	4
87.661	2	$\bar{1}$	1	8	140.949	4	[$\bar{8}$	2	6]
88.029	3	1	1	8	142.117	4	$\bar{5}$	3	4
89.216	3	9	0	1					
89.589		$\bar{3}$	2	5					
89.589	2	5	2	3					
90.409	6	6	2	0					
90.633	4	4	0	8					
91.707	8	0	2	6					
91.927	6	8	1	3					

72-0514 Wavelength= 1.54060 C

VO2

Vanadium Oxide

Rad.: CuKa1 λ: 1.54060 Filter: d-sp: Calculated

Cut off: 17.7 Int.: Calculated I/Icor.: 3.49

Ref: Calculated from ICSD using POWD-12++, (1997)

Ref: Andersson, G., Acta Chem. Scand., 10, 623 (1956)

Sys.: Monoclinic S.G.: $P2_1/c$ (14)

a: 5.743 b: 4.517 c: 5.375 A: 1.2714 C: 1.1899

α: β: 122.610 γ: Z: 4 mp:

Ref: Ibid.

Dx: 4.690 Dm: 4.650

Peak height intensity. R-factor: 0.100. O2 V type. C.D. Cell: a=5.375, b=4.517, c=5.348, β=115.23, a/b=1.1899, c/b=1.1840, S.G.=P21/a(14). PSC: mP12. At least one TF missing. Mwt: 82.94. Volume[CD]: 117.45.

2θ	Int	h	k	l	2θ	Int	h	k	l
18.324	6	1	0	0	55.645	259	2	2	0
26.920	49	$\bar{1}$	1	1	55.645		$\bar{2}$	1	1
26.920		1	1	0	56.954	23	$\bar{1}$	1	3
27.878	999*	0	1	1	57.127	14	3	0	0
33.433	37	$\bar{1}$	0	2	57.127		1	1	2
36.946	120	$\bar{2}$	0	2	57.603	125	0	2	2
37.069	283	$\bar{2}$	1	1	60.657	20	$\bar{3}$	1	3
37.140	254	2	0	0	60.957	3	3	1	0
39.071	9	$\bar{1}$	1	2	64.545	11	$\bar{3}$	2	2
39.209	6	1	1	1	64.894	51	$\bar{4}$	0	2
39.786	30	0	0	2	65.113	46	0	1	3
39.884	44	0	2	0	65.251	36	0	3	1
42.180	102	$\bar{2}$	1	2	66.803	5	$\bar{2}$	2	3
42.353	94	2	1	0	67.056	3	2	2	1
44.181	15	$\bar{1}$	2	1	68.234	3	$\bar{1}$	2	3
44.181		1	2	0	68.390	3	1	2	2
44.809	24	0	2	1	68.523	1	$\bar{4}$	1	2
48.513	15	$\bar{3}$	0	2	70.238	23	$\bar{2}$	0	4
51.434	1	$\bar{2}$	2	1	70.573	75	$\bar{2}$	3	1
52.996	19	$\bar{3}$	1	1	70.573		2	0	2
52.996		$\bar{1}$	2	2	71.448	8	$\bar{3}$	0	4
53.105	24	1	0	2	71.607	7	$\bar{3}$	2	3
53.105		1	2	1	71.925	24	$\bar{4}$	1	3
55.363	95	$\bar{2}$	1	3	71.925	24	1	3	1
55.490	144	$\bar{2}$	2	2	72.170	35	$\bar{4}$	1	1

2θ	Int	h	k	l
73.743	1	$\bar{2}$	1	4
74.116	6	2	1	2
74.116		2	3	0
74.930	1	$\bar{3}$	1	4
75.129	4	$\bar{1}$	0	4
75.381	2	3	1	1
75.739	3	0	2	3
75.820	3	0	3	2
78.759	4	1	1	3
78.952	5	$\bar{4}$	2	2
79.125	4	4	0	0
82.011	4	$\bar{3}$	3	2
82.011		$\bar{4}$	1	4
82.197	3	$\bar{4}$	2	3
82.431	4	$\bar{4}$	2	1
82.431		4	1	0
83.947	3	$\bar{2}$	2	4
84.084	4	$\bar{2}$	3	3
84.297	12	2	2	2
84.297		2	3	1
85.095	8	$\bar{3}$	2	4
85.095		$\bar{5}$	0	2
85.411	6	$\bar{1}$	3	3
85.557	6	3	2	1
85.557		1	3	2
85.769	10	0	0	4
86.021	11	0	4	0
88.334	6	$\bar{5}$	1	3
88.625	11	$\bar{3}$	3	3
88.625		$\bar{1}$	2	4
88.901	11	3	3	0
88.901	11	$\bar{1}$	4	1
89.106	6	0	1	4
89.341	5	0	4	1

87-1120 Wavelength= 1.54060 C

NaK(V2O6)

Sodium Potassium Vanadium Oxide

Rad.: CuKa1 λ: 1.54060 Filter: d-sp: Calculated

Cut off: 17.7 Int.: Calculated I/Icor.: 1.63

Ref: Calculated from ICSD using POWD-12++, (1997)

Ref: Xu, J.-N et al., J. Struct. Chem. (China), 15, 458 (1996)

Sys.: Monoclinic S.G.: C2 (5)

a: 10.568(3) b: 10.082(2) c: 5.81(2) A: 1.0482 C: 0.5763

α: β: 103.870(2) γ: Z: 4 mp:

Ref: Ibid.

Dx: 2.873 Dm: 2.850

Peak height intensity. R-factor: 0.029. PSC: mC40. Mwt: 259.97. Volume[CD]: 600.99.

2θ	Int	h	k	l	2θ	Int	h	k	l	2θ	Int	h	k	l	2θ	Int	h	k	l
12.298	106	1	1	0	36.476	22	0	2	2	47.867	8	$\bar{1}$	1	3	63.182	22	6	2	1
15.698	1	0	0	1	37.029	36	$\bar{2}$	2	2	47.980	23	$\bar{1}$	5	1	64.015	10	$\bar{5}$	5	1
17.272	260	2	0	0	37.188	25	$\bar{3}$	1	2	48.394	42	0	0	3	64.156	23	7	1	0
17.579	167	0	2	0	37.490	52	3	3	0	48.394		0	4	2	64.156	23	$\bar{6}$	0	3
18.271	11	$\bar{1}$	1	1	38.219	19	$\bar{3}$	3	1	48.835	7	$\bar{2}$	4	2	64.501	56	0	6	2
20.402	1	$\bar{2}$	0	1	39.113	227	0	4	1	49.035	42	$\bar{5}$	1	2	64.890	98	$\bar{2}$	6	2
21.578	39	1	1	1	39.200	152	$\bar{4}$	2	1	49.479	2	1	5	1	64.890	98	$\bar{7}$	1	2
23.652	364	0	2	1	39.816	5	2	4	0	50.234	57	$\bar{3}$	1	3	65.192	36	$\bar{1}$	1	4
24.742	290	2	2	0	39.970	4	2	0	2	50.591	18	$\bar{4}$	4	1	65.377	18	1	7	0
27.055	999*	$\bar{2}$	2	1	41.052	46	$\bar{1}$	3	2	50.741	53	$\bar{4}$	4	0	65.918	36	$\bar{3}$	1	4
27.519	293	3	1	0	41.369	33	$\bar{2}$	4	1	50.839	126	$\bar{2}$	2	3	65.918		6	4	0
27.914	19	$\bar{1}$	3	0	41.490	83	$\bar{4}$	0	2	51.322	100	$\bar{5}$	3	1	66.111	31	4	4	2
28.471	284	$\bar{3}$	1	1	43.392	8	3	3	1	51.682	12	5	1	1	66.221	34	0	0	4
31.109	185	$\bar{1}$	3	1	43.972	19	$\bar{2}$	2	2	51.858	9	0	2	3	66.517	33	4	6	0
31.653	793	2	2	1	44.082	27	$\bar{5}$	1	1	51.987	4	$\bar{6}$	0	1	66.517		3	5	2
31.653		0	0	2	44.374	23	1	3	2	52.187	7	5	3	0	67.096	12	$\bar{1}$	7	1
32.057	45	$\bar{1}$	1	2	44.672	32	2	4	1	52.276	5	1	1	3	67.096	12	$\bar{6}$	2	3
32.324	51	$\bar{2}$	0	2	45.051	93	$\bar{5}$	1	0	52.651	22	3	5	0	67.658	3	$\bar{4}$	0	4
33.226	9	1	3	1	45.382	10	$\bar{4}$	2	2	53.212	6	$\bar{3}$	5	1	68.035	23	$\bar{7}$	3	1
34.908	151	3	1	1	45.825	68	1	5	0	53.548	25	6	0	0	68.035		2	4	3
34.908		4	0	0	45.825		4	2	1	53.717	55	4	0	2	68.314	12	1	7	1
35.590	9	0	4	0	47.260	2	3	1	2	54.178	2	3	3	2	68.314		3	3	3
36.080	63	1	1	2	47.260		$\bar{2}$	0	3	54.571	54	0	6	0	68.557	7	4	0	3
										54.571		2	4	2	68.557		5	3	2
										54.728	111	$\bar{1}$	3	3	68.933	6	$\bar{3}$	5	3
										55.310	7	$\bar{6}$	2	1	69.114	3	0	2	4
										55.437	6	$\bar{1}$	5	2	69.752	15	2	6	2
										55.742	29	$\bar{6}$	0	2	69.970	9	7	3	0
										55.742	29	$\bar{5}$	3	2	70.134	10	1	1	4
										55.929	17	2	0	3	70.134		5	5	1
										56.207	32	4	4	1	70.523	3	$\bar{4}$	2	4
										56.808	6	6	2	0	70.744	8	$\bar{7}$	3	2
										56.970	18	4	2	2	70.744		7	1	1
										57.153	10	0	6	1	70.946	5	3	7	0
										57.325	6	3	5	1	70.946	5	$\bar{1}$	3	4
										57.683	35	2	6	0	71.174	2	4	6	1
										58.222	104	5	3	1	71.382	3	4	2	3
										58.905	71	$\bar{3}$	5	2	71.382	3	$\bar{3}$	7	1
										58.905	71	$\bar{6}$	2	2	71.660	3	3	3	4
										59.101	69	2	2	3	71.877	3	6	4	1
										60.567	8	$\bar{2}$	4	3	72.141	11	$\bar{7}$	1	3
										61.478	25	2	6	1	72.141		$\bar{5}$	1	4
										61.478		0	4	3	73.314	10	$\bar{1}$	7	2
										62.132	14	$\bar{7}$	1	1	73.314		$\bar{8}$	0	2
										62.440	11	3	1	3	73.723	15	6	2	2
										62.679	14	5	1	2	73.723		2	0	4

2θ	Int	h	k	l	2θ	Int	h	k	l	2θ	Int	h	k	l
74.149	6	$\bar{8}$	2	1	85.754	6	$\bar{2}$	2	5					
75.055	2	$\bar{2}$	6	3	85.847	6	4	8	0					
75.357	2	0	8	0	86.128	4	0	0	5					
75.604	7	$\bar{6}$	4	3	86.243	9	4	0	4					
75.604	7	1	3	4	86.518	17	1	5	4					
75.877	4	0	6	3	86.518		5	7	1					
76.326	22	7	3	1	86.809	9	7	3	2					
76.326		$\bar{3}$	7	2	87.050	2	1	7	3					
76.508	31	$\bar{6}$	0	4	87.334	1	$\bar{6}$	4	4					
76.508	31	2	2	4	88.006	15	6	2	3					
77.555	12	0	4	4	88.112	18	$\bar{9}$	3	1					
77.555		0	8	1	88.921	3	2	8	2					
77.685	10	$\bar{7}$	3	3	88.921	3	4	2	4					
77.685		$\bar{5}$	3	4	89.057	4	$\bar{2}$	6	4					
78.852	3	$\bar{4}$	4	4	89.057	4	$\bar{6}$	6	3					
79.043	24	$\bar{2}$	8	1										
79.245	13	$\bar{6}$	2	4										
79.680	14	4	4	3										
79.680	14	5	5	2										
79.988	8	6	6	0										
80.127	5	3	1	4										
80.127		$\bar{4}$	6	2										
80.913	22	$\bar{5}$	7	1										
81.042	41	7	5	0										
81.330	27	2	8	1										
81.427	15	7	1	2										
81.600	19	$\bar{5}$	7	0										
81.711	28	$\bar{7}$	5	2										
81.987	14	$\bar{2}$	6	3										
81.987	14	$\bar{1}$	5	4										
82.412	16	$\bar{8}$	4	1										
82.412		$\bar{8}$	2	3										
82.535	15	$\bar{5}$	3	3										
82.654	24	$\bar{3}$	5	4										
83.207	4	$\bar{3}$	7	2										
83.430	4	$\bar{7}$	1	4										
83.659	13	$\bar{1}$	7	3										
84.053	12	$\bar{0}$	8	2										
84.053		$\bar{9}$	1	2										
84.154	12	$\bar{3}$	1	5										
84.385	7	$\bar{2}$	8	2										
84.537	8	$\bar{5}$	7	2										
84.739	4	8	4	0										
85.487	2	3	3	4										
85.487	2	$\bar{6}$	6	1										
85.754	6	$\bar{4}$	8	1										