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A Refinement of the Crystal Structure of $MoOPO_4$

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The structure of $MoOPO_4$ has previously been determined and reported in 1964. Data for the refinement was collected by counter-technique using an automatic General Electric single-crystal diffractometer. The result of the least-squares refinement is in good agreement with the original parameter determination but much lower standard deviation in the parameters has been achieved.

The crystal structure of $MoOPO_4$ was reported some years ago by Kierkegaard and Westerlund.[1] The tetragonal structure (space group $P4/n$) was derived on the basis of three-dimensional X-ray film data taken with CuK radiation. The parameters were refined to moderate accuracy using the least-squares method. The structure was described in terms of distorted molybdenum-oxygen octahedra joined by sharing corners to form chains parallel to the c axis. These chains are connected by PO_4 tetrahedra so that each MoO_6 octahedra is sharing corners with four phosphate tetrahedra and each PO_4 tetrahedron with four MoO_6 octahedra, thus giving a three-dimensional network. As pointed out by Eick and Kihlborg [2] the structure may, alternatively, be described in terms of a slightly distorted cubic close-packed arrangement of oxygen atoms in which 1/5 of the octahedral holes are occupied by molybdenum atoms and 1/10 of the tetrahedral holes by phosphorus atoms.

Later on the compounds $NbOPO_4$ [3] and $VOMoO_4$ [2] were found to be isostructural with $MoOPO_4$. Similarities were also found between the structures of the latter and the orthorhombic phase of $VOSO_4$.[4] Recently it has been reported by Ladwig [5a] and also by Longo and Arnott [5b] that the structure of tetragonal $VOSO_4$ is of the $MoOPO_4$ type.

In order to obtain a higher accuracy in the structural details of the $MoOPO_4$ structure a refinement based on diffractometer data was undertaken. This article will describe the results thus obtained.

A single crystal—a well shaped plate with the dimensions 0.064 mm (in the direction of the a axis) $\times$ 0.064 mm (b) $\times$ 0.020 mm (c)—was mounted on a

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General Electric XRD-5 Diffractometer equipped with a scintillation detector and a pulse-height analyzer. Nb-filtered MoK radiation was used and the pulse-height analyzer was set to collect about 90 % of the $K\alpha$ radiation. The $\theta-2\theta$ scanning technique was used to measure 1071 reflections with $2\theta \leq 100°$, 769 of which were observable. Each reflection was scanned twice at a rate of 1°/min through the scan interval according to the formula: $\Delta 2\theta = a + b \cdot \tan\theta$ where $a = 1.4$ and $b = 2.6$. A 100-second background count was collected at each end of the scan range. Lp and absorption correction ($\mu = 42.8$ cm^{-1}) were applied on the net intensity counts. Corrections for secondary extinction effects were applied according to the formula given by Zachariasen.[6] The value of the constant c in the formula obtained for this crystal of $MoOPO_4$ was $(0.465 \pm 0.013) \times 10^{-2}$.

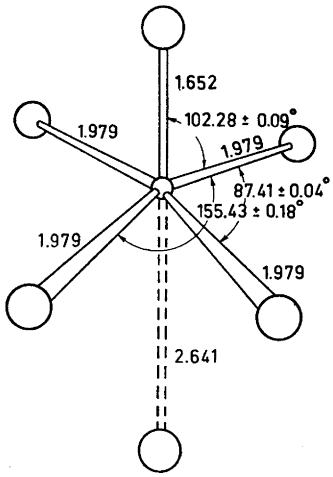

Fig. 1. The coordination of oxygen atoms (large circles) around the molybdenum (small circle). Angles (and their estimated standard deviations) related to the others by symmetry have not been indicated.

Table 1a. Atomic coordinates and standard deviations (σ) obtained in the final cycle of the least-squares refinement of $MoOPO_4$.

Atom	$x \pm 10^5\ \sigma(x)$	$y \pm 10^5\ \sigma(y)$	$z \pm 10^5\ \sigma(z)$
Mo	1/4	1/4	0.80244 ± 13
P	1/4	3/4	1/2
O_1	1/4	1/4	0.18726 ± 124
O_2	0.80876 ± 53	0.44256 ± 49	0.29562 ± 72

Table 1b. Anisotropic thermal parameters (Å^2) with their standard deviations (σ) obtained in the final cycle of the least-squares refinement of $MoOPO_4$.

$(T = \exp[\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl])$

Atom	$10^5\ \beta_{11}$	$10^5\ \beta_{22}$	$10^5\ \beta_{33}$	$10^5\ \beta_{12}$	$10^5\ \beta_{13}$	$10^5\ \beta_{23}$
Mo	220 ± 8	220 ± 8	735 ± 21	0	0	0
P	262 ± 17	262 ± 17	862 ± 59	0	0	0
O_1	476 ± 49	476 ± 49	910 ± 142	0	0	0
O_2	713 ± 49	528 ± 44	1456 ± 99	131 ± 79	195 ± 115	409 ± 112

Table 2. Observed and calculated structure factors for $MoOPO_4$.

H	K	L	KFOB	FCAL
0	1	1	80.60	67.80
0	1	2	21.45	22.19
0	1	3	27.68	27.35
0	1	4	27.47	27.02
0	1	6	17.72	18.42
0	1	7	11.42	12.09
0	2	0	110.63	102.06
0	2	2	31.29	30.64
0	2	3	56.79	56.79
0	2	4	20.74	21.03
0	2	5	21.81	22.84
0	2	6	18.50	18.41
0	2	7	20.71	20.48
0	2	8	10.65	10.45
0	3	1	60.70	60.30
0	3	2	14.74	15.14
0	3	3	29.87	29.25
0	3	4	21.20	20.62
0	3	6	13.70	14.58
0	3	7	13.06	12.10
0	4	0	80.14	76.91
0	4	2	26.64	25.84
0	4	3	46.07	46.47
0	4	4	18.98	18.83
0	4	5	19.34	19.70
0	4	6	17.05	17.17
0	4	7	18.47	18.35
0	4	8	10.65	10.26
0	5	1	22.52	22.89
0	5	2	26.59	26.54
0	5	3	10.02	12.01
0	5	4	32.01	31.95
0	5	6	21.40	21.60
0	6	0	53.86	54.91
0	6	2	18.50	18.70
0	6	3	39.29	38.38
0	6	4	16.53	15.90
0	6	5	17.55	18.18
0	6	6	15.27	13.94
0	6	7	17.05	16.19
0	7	1	27.06	26.73
0	7	2	16.89	17.20
0	7	3	13.06	13.57
0	7	4	22.45	20.22
0	7	6	15.65	15.89
0	8	0	30.80	31.74
0	8	2	8.25	7.34
0	8	3	30.10	26.74
0	8	4	10.11	9.48
0	8	5	21.40	20.72
0	8	6	12.15	11.23
0	9	1	19.26	19.05
0	9	2	15.37	15.20
0	9	3	10.11	10.06
0	9	4	20.57	19.72
0	10	0	33.08	36.30
0	10	2	14.39	12.34
0	10	3	22.83	23.04
0	10	4	9.61	9.24
0	11	1	12.15	13.30
0	11	2	12.40	11.97
0	11	3	8.25	6.47
0	12	0	21.52	21.71
1	0	1	77.06	67.80
1	0	2	21.31	22.19
1	0	3	26.80	27.35
1	0	4	26.62	27.02
1	0	6	17.22	18.42
1	0	7	12.60	12.09
1	1	0	44.33	42.34
1	1	1	56.29	53.53
1	1	2	59.69	58.64
1	1	3	34.72	34.55
1	1	5	45.71	46.22
1	1	7	12.60	11.37
1	1	8	18.81	18.17
1	2	1	19.45	11.21
1	2	2	40.02	41.93
1	2	3	11.44	8.60
1	2	4	41.06	42.83
1	2	6	26.73	27.32
1	2	7	10.11	6.64
1	2	8	13.15	10.20
1	3	0	49.72	41.34
1	3	1	38.10	40.07
1	3	2	55.55	54.43
1	3	3	25.35	27.62
1	3	5	40.02	41.34
1	3	7	8.92	9.82
1	3	8	17.55	17.44
1	4	1	35.43	32.82
1	4	2	24.28	24.68
1	4	3	16.72	16.00
1	4	4	28.66	29.76
1	4	6	19.86	20.26
1	4	7	9.82	9.92
1	4	8	9.23	7.59
1	5	0	19.60	15.36
1	5	1	33.48	34.13
1	5	2	35.39	33.43
1	5	3	27.40	29.68
1	5	5	41.12	42.09
1	5	7	12.84	12.35
1	6	1	31.92	28.89
1	6	2	18.09	19.70
1	6	3	16.65	14.97
1	6	4	23.21	24.87
1	6	6	15.46	17.01
1	6	7	7.52	8.37
1	7	0	30.44	29.81
1	7	1	20.86	21.15
1	7	2	39.63	39.19
1	7	3	13.27	14.20
1	7	5	26.28	26.76
1	8	1	21.66	21.50
1	8	2	16.63	16.35
1	8	3	8.92	10.77
1	8	4	21.52	21.36
1	8	6	14.10	15.18
1	9	0	20.12	21.00
1	9	1	16.70	16.81
1	9	2	29.11	28.69
1	9	3	13.06	13.40
1	9	5	22.45	22.70
1	10	1	26.14	25.02
1	10	2	8.99	8.55
1	10	3	14.51	13.33
1	10	4	10.92	11.18
1	11	0	15.27	16.78
1	11	1	12.84	12.03
1	11	2	21.63	21.60
1	11	3	10.37	9.92
1	12	1	16.01	14.81
2	0	0	111.47	102.06
2	0	2	31.30	30.64
2	0	3	56.91	56.79
2	0	4	21.02	21.03
2	0	5	22.59	22.84
2	0	6	18.19	18.41
2	0	7	20.14	20.48
2	0	8	10.92	10.45
2	1	1	40.74	45.91
2	1	2	28.20	27.00
2	1	3	19.03	20.81
2	1	4	32.18	30.88
2	1	6	21.24	20.57
2	1	7	10.65	10.89
2	2	0	65.67	67.23
2	2	1	9.23	9.11
2	2	2	15.70	15.03
2	2	3	62.24	61.69
2	2	4	16.36	16.86
2	2	5	27.06	27.69
2	2	6	17.86	17.27
2	2	7	22.67	22.76
2	2	8	7.62	8.71
2	3	1	46.29	49.58
2	3	2	19.93	19.93
2	3	3	21.71	23.47
2	3	4	25.19	24.89
2	3	6	16.17	16.90
2	3	7	10.65	10.81
2	4	0	82.87	85.26
2	4	2	31.34	33.28
2	4	3	40.48	37.92
2	4	4	15.43	20.73
2	4	5	14.70	14.22
2	4	6	18.03	17.29
2	4	7	16.01	15.23
2	5	1	32.44	33.06
2	5	2	19.88	19.75
2	5	3	14.70	16.47
2	5	4	25.59	25.03
2	5	6	16.70	17.34
2	5	7	9.23	9.53
2	6	0	59.31	58.65
2	6	2	23.35	22.39
2	6	3	34.46	34.60
2	6	4	16.89	16.82
2	6	5	15.27	14.86
2	6	6	11.68	13.92
2	6	7	14.32	14.13
2	7	1	37.15	37.89
2	7	2	10.37	10.61
2	7	3	19.69	19.75
2	7	4	14.10	14.13
2	7	6	8.92	9.75
2	8	0	41.43	41.77
2	8	2	14.89	15.27
2	8	3	29.23	28.93
2	8	4	10.65	11.84
2	8	5	14.32	14.11
2	8	6	13.06	11.95
2	9	1	22.43	21.45
2	9	2	13.06	13.31
2	9	3	11.91	11.45
2	9	4	16.01	17.27
2	10	0	36.53	34.96
2	10	2	15.82	15.01
2	10	3	19.55	20.41
2	10	4	12.60	10.52
2	11	1	16.01	16.06
2	11	2	12.84	10.04
2	12	0	19.69	19.56
3	0	1	69.80	66.30
3	0	2	15.13	15.14
3	0	3	28.47	29.25
3	0	4	21.65	20.82
3	0	6	14.51	14.58
3	0	7	12.39	12.10
3	1	0	75.20	77.98
3	1	1	30.80	30.36
3	1	8	20.57	20.45
3	2	1	43.00	42.02
3	2	2	22.17	22.82
3	2	3	19.31	19.84
3	2	4	26.21	27.75
3	2	6	16.53	18.88
3	2	7	10.92	10.62
3	2	8	6.58	6.86
3	3	0	49.68	44.93
3	3	1	29.25	30.20
3	3	2	58.55	58.11
3	3	3	17.72	17.85
3	3	5	32.89	33.54
3	4	1	46.61	49.33
3	4	2	16.20	13.40
3	4	3	23.00	23.97
3	4	4	21.00	18.28
3	4	6	13.70	12.89
3	4	7	12.35	11.39
3	5	0	25.87	26.70
3	5	1	27.09	27.31
3	5	2	39.26	39.51
3	5	3	21.12	20.71
3	5	5	34.79	34.20
3	5	7	9.23	8.97
3	6	1	25.04	24.89
3	6	2	19.55	19.58
3	6	3	13.06	13.00
3	6	4	25.47	24.85
3	6	6	16.36	17.09
3	6	7	9.23	7.57
3	7	0	25.02	26.67
3	7	1	20.00	20.10
3	7	2	34.86	35.93
3	7	3	14.89	14.37
3	7	5	27.25	25.84
3	8	1	19.41	19.24
3	8	2	17.53	16.01
3	8	3	7.52	9.54
3	8	4	21.03	20.97
3	8	6	15.84	15.00
3	9	0	12.37	12.39
3	9	1	18.05	17.83
3	9	2	21.24	21.24
3	9	3	17.69	17.46
3	9	5	26.73	26.03
3	10	1	18.03	17.99
3	10	2	9.82	11.39
3	10	3	8.25	9.57
3	10	4	13.91	14.86
3	11	0	13.27	13.50
3	11	1	12.60	12.01
3	11	2	19.12	18.52
3	11	3	10.65	11.03
4	0	0	78.29	76.91
4	0	2	26.52	25.83
4	0	3	45.92	46.47
4	0	4	18.98	18.83
4	0	5	18.81	19.70
4	0	6	17.55	17.17
4	0	7	18.67	18.35
4	0	8	10.92	10.26
4	1	1	44.65	46.46
4	1	2	19.62	18.31
4	1	3	20.88	21.80
4	1	4	23.73	23.60
4	1	6	16.89	16.44
4	1	7	10.92	11.20
4	2	0	58.98	52.40
4	2	2	19.22	11.65
4	2	3	50.47	52.96
4	2	4	14.89	14.26
4	2	5	24.09	25.78
4	2	6	15.46	16.13
4	2	7	21.40	21.51
4	3	1	29.18	23.23
4	3	2	23.76	26.34
4	3	3	14.10	12.14
4	3	4	30.06	31.75
4	3	6	20.14	21.48
4	3	7	8.25	8.18
4	4	0	58.36	57.45
4	4	2	19.12	19.12
4	4	3	40.07	40.59
4	4	4	14.89	15.89
4	4	5	16.34	18.63

Table 2. Continued.

H	K	L	KFOB	FCAL
4	4	6	15.46	14.92
4	4	7	15.84	16.84
4	5	1	24.80	20.92
4	5	1	24.80	20.92
4	5	2	20.86	22.05
4	5	3	10.92	10.41
4	5	4	27.59	27.93
4	5	6	19.72	19.53
4	5	7	9.23	7.84
4	6	0	39.60	36.69
4	6	2	12.15	8.92
4	6	3	38.95	39.92
4	6	4	10.65	11.15
4	6	5	21.40	21.57
4	6	6	13.91	11.91
4	7	1	26.04	25.85
4	7	2	15.65	14.51
4	7	3	14.29	13.62
4	7	4	18.03	18.80
4	7	6	12.84	12.95
4	8	0	36.74	36.14
4	8	2	11.91	12.77
4	8	3	27.13	27.45
4	8	4	7.90	10.17
4	8	5	13.89	14.10
4	9	1	17.53	16.78
4	9	2	13.48	13.67
4	9	3	9.82	8.61
4	9	4	19.12	17.94
4	10	0	31.34	29.96
4	10	2	13.48	13.14
4	10	3	19.41	19.56
4	10	4	9.23	9.46
4	11	1	19.69	18.27
5	0	1	22.12	22.89
5	0	2	25.78	26.54
5	0	3	11.68	12.01
5	0	4	32.27	31.95
5	0	6	21.93	21.60
5	0	7	8.25	8.11
5	1	0	32.53	35.24
5	1	1	28.87	28.97
5	1	2	45.47	47.07
5	1	3	19.88	20.07
5	1	5	36.20	34.31
5	1	7	7.90	7.96
5	2	1	26.04	24.80
5	2	2	22.50	23.95
5	2	3	11.91	12.65
5	2	4	28.97	29.52
5	2	6	19.26	20.21
5	2	7	8.25	8.41
5	3	0	22.50	22.66
5	3	1	28.09	28.81
5	3	2	36.96	37.08
5	3	3	22.47	23.44
5	3	5	36.10	36.11
5	3	7	9.51	9.60
5	4	1	35.84	36.92
5	4	2	15.84	14.53
5	4	3	18.19	19.16
5	4	4	20.43	18.92
5	4	6	13.70	12.90
5	4	7	9.82	9.35
5	5	0	29.16	29.00
5	5	1	20.71	21.41
5	5	2	39.07	38.62
5	5	3	13.89	14.74
5	5	5	26.49	27.22
5	6	1	26.49	24.88
5	6	2	14.51	15.80
5	6	3	14.29	13.00
5	6	4	20.43	20.45
5	6	6	13.91	14.20
5	7	0	27.23	28.34
5	7	1	16.53	16.29
5	7	2	35.27	35.24
5	7	3	9.82	10.31
5	7	5	21.12	20.43
5	8	1	27.44	26.28
5	8	2	8.25	9.61
5	8	3	13.48	13.74
5	8	4	11.91	12.64
5	9	0	18.17	17.12
5	9	1	14.89	14.04
5	9	2	23.07	23.22
5	9	3	10.11	12.01
5	10	1	19.55	18.84
5	10	2	7.52	8.97
5	10	3	9.82	10.24
5	11	0	17.03	16.47
5	11	1	10.37	9.64
6	0	0	53.96	54.91
6	0	2	18.50	18.70
6	0	3	39.67	39.38
6	0	4	17.05	15.90
6	0	5	17.55	18.18
6	0	6	14.32	13.94
6	0	7	14.70	16.15
6	1	1	42.28	43.95
6	1	2	13.06	12.11
6	1	3	20.86	22.52
6	1	4	17.38	16.22
6	1	6	9.82	11.10
6	1	7	9.82	10.37
6	0	7	15.46	15.54
6	0	8	14.70	13.86
6	2	0	59.76	59.57
6	2	2	23.23	22.76
6	2	3	33.27	33.65
6	2	4	16.36	16.38
6	2	5	13.06	14.31
6	2	6	16.01	14.67
6	2	7	12.39	14.14
6	3	1	29.58	30.04
6	3	2	17.39	16.77
6	3	3	14.70	15.50
6	3	4	21.99	21.60
6	3	6	15.65	14.94
6	3	7	9.23	8.55
6	4	0	54.29	56.27
6	4	2	22.59	23.33
6	4	3	29.75	28.48
6	4	4	16.89	16.14
6	4	5	13.70	11.37
6	4	6	13.48	13.33
6	5	1	30.32	30.65
6	5	2	12.60	12.31
6	5	3	16.01	15.70
6	5	4	17.22	16.30
6	5	6	11.91	11.51
6	6	0	35.86	36.39
6	6	2	12.15	11.88
6	6	3	30.70	30.62
6	6	4	10.37	10.48
6	6	5	16.89	16.12
6	6	6	10.92	11.17
6	7	1	20.99	22.48
6	7	2	14.29	12.65
6	7	3	11.42	11.94
6	7	4	17.86	16.44
6	8	0	33.41	32.79
6	8	2	13.06	12.69
6	8	3	21.52	22.70
6	9	1	13.70	13.10
6	9	2	14.29	12.67
6	10	0	20.98	21.81
7	0	1	26.64	26.73
7	0	2	17.38	17.20
7	0	3	13.27	13.57
7	0	4	22.71	22.22
7	0	6	15.08	15.59
7	1	0	30.65	31.90
7	1	1	20.29	20.63
7	1	2	40.55	40.77
7	1	3	12.60	13.09
7	1	5	24.92	25.73
7	2	1	29.75	27.81
7	2	2	14.70	15.44
7	2	3	14.32	13.94
7	2	4	18.50	20.23
7	2	6	12.15	14.39
7	3	0	17.22	15.98
7	3	1	22.45	22.90
7	3	2	27.92	27.78
7	3	3	20.57	20.47
7	3	5	31.23	31.59
7	4	1	22.71	22.81
7	4	2	15.08	16.02
7	4	3	12.39	11.86
7	4	4	19.26	20.76
7	4	6	15.08	14.46
7	5	0	21.52	19.68
7	5	1	18.19	18.43
7	5	2	30.61	28.38
7	5	3	14.32	15.17
7	5	5	24.80	25.31
7	6	1	16.01	14.35
7	6	2	17.05	16.68
7	6	3	7.52	7.03
7	6	4	21.81	21.87
7	7	0	12.15	14.61
7	7	1	15.82	16.06
7	7	2	21.24	22.17
7	7	3	13.70	15.01
7	8	1	19.99	18.22
7	8	2	11.68	10.58
7	8	3	11.18	9.55
7	8	4	13.70	13.87
7	9	0	11.18	12.29
7	9	1	11.42	12.42
7	9	2	18.64	17.64
7	9	3	11.42	11.95
8	0	0	30.61	31.94
8	0	3	37.62	36.74
8	0	4	8.58	9.40
8	0	5	22.33	20.72
8	0	6	12.15	11.23
8	1	1	21.12	22.09
8	1	2	15.46	16.46
8	1	3	11.91	11.52
8	1	4	21.93	21.30
8	1	6	15.46	14.82
8	2	0	38.72	38.63
8	2	2	14.89	13.02
8	2	3	30.42	31.01
8	2	4	10.37	11.27
8	2	5	16.20	15.92
8	2	6	11.42	11.40
8	3	1	15.46	16.69
8	3	2	19.26	17.74
8	3	4	24.19	23.01
8	3	6	14.89	16.23
8	4	0	35.29	35.44
8	4	2	13.70	12.62
8	4	3	27.37	28.51
8	4	4	10.65	10.97
8	4	5	15.65	14.68
8	5	1	24.54	24.01
8	5	2	11.68	11.12
8	5	3	12.60	12.75
8	5	4	13.48	14.47
8	6	0	31.27	30.84
8	6	2	12.15	11.28
8	6	3	23.47	24.12
8	6	4	9.82	9.00
8	7	1	18.64	18.93
8	7	2	9.82	10.49
8	7	3	9.82	10.24
8	7	4	14.51	13.63
8	8	0	33.79	30.96
8	8	2	16.17	14.07
8	8	3	15.65	16.33
8	8	1	17.22	16.24
9	0	1	18.17	19.06
9	0	2	15.46	15.20
9	0	3	8.92	10.66
9	0	4	19.41	19.72
9	1	0	22.67	22.86
9	1	1	15.82	16.39
9	1	2	30.42	30.22
9	1	3	10.65	12.41
9	1	5	21.57	21.62
9	2	1	27.47	27.47
9	2	2	9.23	9.93
9	2	3	15.08	14.70
9	2	4	11.91	12.91
9	3	0	18.95	20.37
9	3	1	16.53	15.52
9	3	2	25.47	27.25
9	3	3	12.39	12.28
9	3	5	22.59	21.17
9	4	1	22.45	22.64
9	4	2	9.82	10.67
9	4	3	12.33	12.14
9	4	4	15.08	13.88
9	4	6	23.81	23.51
9	5	1	11.42	12.25
9	5	2	28.51	28.18
9	6	1	19.03	18.60
9	6	2	10.43	9.62
9	6	3	9.82	9.52
9	7	0	14.26	14.57
9	7	1	12.60	11.87
9	7	2	20.43	19.50
9	7	3	11.68	10.63
9	8	1	17.53	16.47
10	0	0	33.63	32.30
10	0	2	12.39	12.34
10	0	3	22.71	23.04
10	0	4	10.37	9.24
10	1	1	23.83	21.12
10	1	2	9.82	10.64
10	1	3	10.65	11.08
10	1	4	14.70	13.96
10	2	0	26.27	34.83
10	2	2	15.82	14.74
10	2	3	18.95	20.12
10	2	4	10.11	9.91
10	3	1	17.05	16.92
10	3	2	11.18	11.48
10	3	3	7.50	8.42
10	3	4	16.20	15.25
10	4	0	26.27	25.33
10	4	2	9.51	8.62
10	4	3	23.47	22.92
10	4	4	8.25	7.34
10	5	1	17.22	16.64
10	5	2	10.11	9.84
10	5	3	9.82	8.65
10	6	0	21.99	21.89
11	0	1	13.06	12.30
11	0	2	12.84	11.97
11	0	3	8.25	6.47
11	1	0	10.92	11.47
11	1	1	13.89	13.64
11	1	2	15.82	17.61
11	1	3	12.84	13.56
11	2	1	17.05	15.76
11	2	2	10.92	10.41
11	2	3	7.90	8.22
11	3	0	10.92	11.78
11	3	1	12.60	12.60
11	3	2	16.35	17.28
11	3	3	13.89	12.33
11	4	1	10.65	11.58
11	4	2	9.82	11.42
11	5	0	14.70	13.89
11	5	1	11.68	10.54
12	0	0	21.24	21.71
12	1	1	11.91	13.04
12	2	0	25.47	25.18
0	0	1	11.77	12.49
0	0	2	71.92	67.01
0	0	3	39.60	41.00
0	0	4	28.75	28.71
0	0	5	12.39	12.04
0	0	6	20.29	20.50

The refinement of the parameters was performed by means of a full-matrix least-squares program. The parameters derived from film data were starting values of the coordinates and the individual anisotropic temperature factor. Using a total of 616 reflections (data with $2<F_{obs}/F_{calc}<0.5$ were given a weight of zero in the least squares refinement) the parameters were refined until the shifts were less than 3 % of their standard deviations. The discrepancy index, R, defined in the usual way and including observed reflections only, then equalled 0.045.

The scattering factor curves used for oxygen and phosphorus were those given by Freeman [7,8] and the curve for molybdenum was that reported by Thomas and Umeda.[9] The real part of the dispersion correction [10] was applied to the scattering factor curves.

The parameters obtained from the last cycle of the refinement are listed in Table 1. Observed and calculated structure factors are listed in Table 2. Interatomic distances and standard deviations determined are presented in Table 3.

Table 3. Interatomic distances (Å) and standard deviations ($\pm\sigma$) in $MoOPO_4$.

Mo—O:		
	Mo—2 O_1 (O_1—2 Mo)	= 1.652±5; 2.641±5
	Mo—4 O_2 (O_2—Mo)	= 1.979±3
P—O:		
	P—4 O_2 (O_2—P)	= 1.522±3
O—O:		
	O_1—12 O_2	= 4×(2.835±5)
		= 4×(2.944±5)
		= 4×(3.010±3)
	O_2—6 O_1	= 2×(2.835; 2.944; 3.010)
	O_2—6 O_2	= 2×(2.484±5) [a]
		= 2.487±6 [a]
		= 2×(2.734±4)
		= 3.027±6

[a] Denote O—O distances within the PO_4 polyhedron.

The present investigation has not changed the general picture of the structure reported in Ref. 1 but given a substantial improvement of the atomic parameters.

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