Topotactic Decomposition and Crystal Structure of White Molybdenum Trioxide–Monohydrate: Prediction of Structure by Topotaxy

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Single crystals of the white $MoO_3 \cdot H_2O$ modification ("α-molybdic acid") were transformed by heating to 160°C into perfect pseudomorphs built up from oriented MoO_3 crystallites of known structure. From the mutual orientation relationship of the unit cells of both phases involved in this topotactic reaction, as determined by X-ray photographs, a model for the so far unknown crystal structure of white $MoO_3 \cdot H_2O$ could be deduced.

Independently, this structure was determined by X-ray diffractometer data then: space group $P\bar{1}$, $a = 7.388$, $b = 3.700$, $c = 6.673$ Å, $\alpha = 107.8$, $\beta = 113.6$, $\gamma = 91.2°$, $Z = 2$. The structure was solved from the Patterson function and refined until $R = 0.088$. It is built up from isolated double chains of strongly distorted $[MoO_5(H_2O)]$-octahedra sharing two common edges with each other.

This result agrees well with the model derived from topotaxy, and it becomes evident how the MoO_3 lattice is formed through corner linking of the isolated double chains after the water molecules are removed. The study of topotactic phenomena seems rather generally applicable to deduce the main features of structures involved and for better understanding of structural relationships.

Introduction

In an earlier investigation, the topotactic dehydrations of molybdenum trioxide–dihydrate and the yellow, monoclinic form of molybdenum trioxide–monohydrate have been followed by thermogravimetry, X-ray diffraction, infrared spectroscopy and scanning electron microscopy (*1*). It has been pointed out that another, white form of molybdenum trioxide–monohydrate is described in literature (*2, 3*), called α-$MoO_3 \cdot H_2O$ when crystallized in well-shaped needles and β-$MoO_3 \cdot H_2O$ when the needle shape is less pronounced. As X-ray data of the "β-form" are missing, it may be suspected that it represents a microcrystalline variety of α-$MoO_3 \cdot H_2O$.

Experimental Methods

For the preparation of $MoO_3 \cdot 2H_2O$, molybdenum trioxide has been dissolved in concentrated hydrochloric acid, diluted and been kept for several days, following the preparation described by Carpéni (*4*). One batch of crystals of $MoO_3 \cdot 2H_2O$ was kept under the mother liquor for about three months, when the growth of bushels of white crystals could be observed (scanning electron micrograph see Fig. 1). These crystals have been identified as molybdenum trioxide-monohydrate by thermogravimetrical determination of the weight loss (Mettler Thermoanalyzer TA 1) and powder X-ray diffraction (Guinier–de Wolff camera, CuK_α radiation) of the product of dehydration, anhydrous MoO_3. They obviously represent the white, needle-shaped α-modification of $MoO_3 \cdot H_2O$. Single crystal X-ray diffraction patterns, although of quite poor quality, could be recorded by means of Buerger Precession and of Weissenberg cameras.

Fig. 1. Needles [010] of white molybdenum trioxide-monohydrate. Scanning electron micrograph (×272).

Crystallographic Data

Weissenberg photographs of various isolated single crystals show sharp, but elongated reflexions with a length of 3–30 mm, indicating "single crystals" built up from tilted subneedles (angle of tilt 6–60°). Nevertheless, the unit cell of the crystals could be determined: Triclinic, $a = 7.388$ Å, $b = 3.700$ Å, $c = 6.673$ Å, $\alpha = 107.8°$, $\beta = 113.6°$, $\gamma = 91.2°$; b being parallel to the needle axis.

Topotactic Relations and Proposal of Crystal Structure

An oriented crystal has been decomposed on the goniometer head, without altering its orientation, during 3–4 hr at 160°C. Weissenberg photographs of the product show it to be anhydrous MoO_3, with the following crystallographic orientation relations (real lattice):

$(001)_{MoO_3 \cdot H_2O} \| (100)_{MoO_3}$ and

$[010]_{MoO_3 \cdot H_2O} \| [001]_{MoO_3}$ (needle axis),

the product remaining well oriented with respect to the educt, i.e., forming pseudomorphs as a consequence of a high degree of topotaxy.

A comparison of the unit cells of $MoO_3 \cdot H_2O$ and of MoO_3 (5, 6) shows that the length of the needle axis is altered only very slightly from $[010]_{MoO_3 \cdot H_2O} = 3.700$ Å to $[001]_{MoO_3} =$
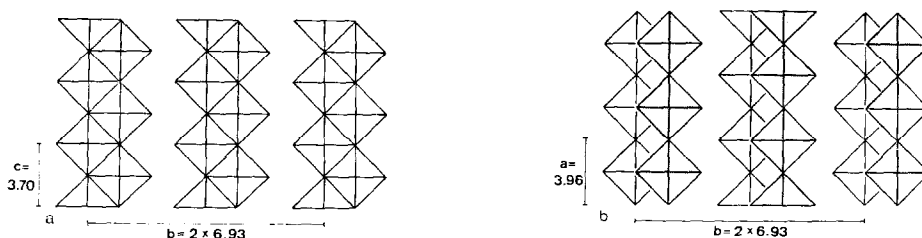
FIG. 2. Idealized drawings of the MoO₃ structure (unit cell dimensions in Å). (a) Plane $(100)_{MoO_3}$ with edge linked octahedral double chains. (b) Plane $(001)_{MoO_3}$. The octahedral double chains [001] from Fig. 2a are linked in [100] through common corners.

3.696 Å, and that the plane $(001)_{MoO_3 \cdot H_2O}$ with $2a = 14.776$, $b = 3.700$ Å and $\gamma = 91.2°$ also remains almost unaffected by the decomposition reaction, leading to $(100)_{MoO_3}$ with $b = 13.855$, $c = 3.696$ Å, and $\alpha = 90.0°$. However, the third axis changes considerably from $[001]_{MoO_3 \cdot H_2O} = 6.673$ Å, $\alpha_{MoO_3 \cdot H_2O} = 107.8$, $\beta_{MoO_3 \cdot H_2O} = 113.6°$ to $[100]_{MoO_3} = 3.963$ Å, $\beta = 90.0$ and $\gamma = 90.0°$.

To get an idea of the unknown crystal structure of white $MoO_3 \cdot H_2O$, the structure of MoO_3 (5, 6) has to be studied in the relevant orientations (Figs. 2a, 2b).

The planar arrays of edge and corner sharing octahedra extend perpendicular to $[010]_{MoO_3}$. As the topotactic relations show that the (100) plane of MoO_3 corresponds closely to (001) of the monohydrate, it may be assumed that the atomic arrangement within this plane is already preformed in the hydrate structure. On the other hand, the [001] axis of the monohydrate differs considerably from $[100]_{MoO_3}$ in direction as well as in length, indicating that this distance is most affected by the loss of

water. Based on these arguments, a crystal structure may be proposed for $MoO_3 \cdot H_2O$, as represented in Figs. 3a, and 3b.

This structure does no longer consist of planes of edge and corner sharing octahedra, but of linear double rows of edge sharing octahedra $[MoO_5(H_2O)]$ parallel to $[010]_{MoO_3 \cdot H_2O}$, being responsible for the tilting disorder in the crystals, as these rows are supposed to be held together in [100] and [001] only by packing and van der Waals forces and most probably by hydrogen bonds.

The chain-like structure proposed is in good agreement with the fibrous morphology observed and with the scanning electron microscopical result that the water leaves the dehydrating crystals through cracks parallel to the needle axis, but otherwise unoriented, whereas the prism faces of the needles remain rather unaffected (Fig. 4).

After yellow $MoO_3 \cdot H_2O$, the here described white modification represents a second case, where the proposal of a crystal structure has been made possible by considerations on
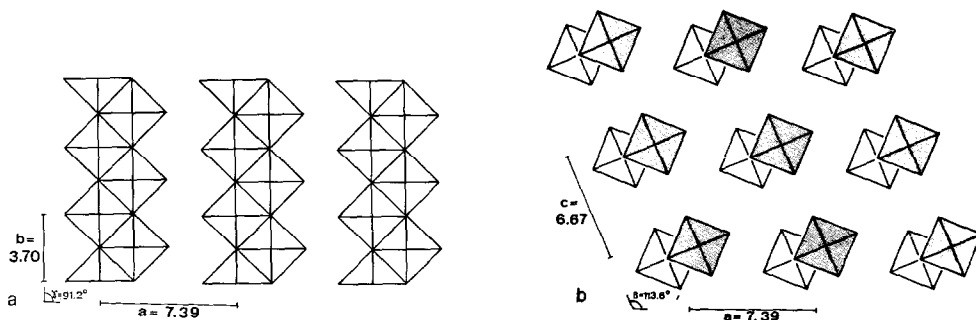
FIG. 3. Idealized model for the crystal structure of white $MoO_3 \cdot H_2O$ as derived from topotaxy (unit cell dimensions in Å). (a) Plane $(001)_{MoO_3 \cdot H_2O}$, to be compared with Fig. 2a. (b) Plane $(010)_{MoO_3 \cdot H_2O}$, to be compared with Fig. 2b.

Fig. 4. Scanning electron micrograph of molybdenum trioxide, pseudomorphous after white molybdenum trioxide–monohydrate. Cracks are running parallel to $[010]_{MoO_3 \cdot H_2O}$ (×1440).

topotaxy. In order to test the validity of these arguments, special attempts have been made to find a single crystal of white $MoO_3 \cdot H_2O$ with as small tilting disorder as possible, suitable to undertake a complete crystal structure determination by X-rays.

Crystal Structure Determination

A large number of needles of $MoO_3 \cdot H_2O$ were examined by Weissenberg photographs. Finally, a very thin needle with approximate dimensions $0.17 \times 0.02 \times 0.02$ mm³ could be

found, which showed a relatively small tilting disorder compared to other ones. This crystal was selected for data collection on a computer controlled Picker four-circle diffractometer (FACS-1 system). The intensity data were measured with MoK_α-radiation by a θ–2θ scan over a relatively large 2θ range of 3° (plus the α_1–α_2 dispersion) with a scan speed of 0.5°/min. Background counts were taken for 40 sec at either end of the 2θ scan for each reflexion. Crystal decomposition and instrument stability were monitored by measuring three standard reflexions after every 12 reflexions.

TABLE I

ATOMIC POSITIONAL COORDINATES IN WHITE
MoO₃·H₂O[a]

Atom	x	y	z
Mo	0.2061(5)	−0.2133(10)	0.0422(6)
O(1)	0.453(4)	−0.119(10)	0.198(5)
O(2)	−0.128(4)	−0.299(8)	−0.052(5)
O(3)	0.181(5)	−0.358(9)	−0.228(5)
O(4)	0.195(5)	0.007(9)	0.409(5)

[a] Standard deviations in the last digit calculated from final least-squares refinement are given in parentheses.

Intensities were collected up to $2\theta \leqslant 40°$, and the standard reflexions were used for an internal scaling of the 747 reflexions measured (including standards and Friedels pairs); 295 reflexions remained after averaging equivalent reflexions and 229 were judged to be observed according to the criterion $I \geqslant 3\sigma(I)$, $\sigma(I)$ being the estimated standard deviation based on counting statistics. The agreement index between the equivalent Friedels pairs, $\sum (|I - \bar{I}|)/\sum (\bar{I})$, was 0.053. Absorption corrections were made using a linear absorption coefficient of $\mu = 39.4$ cm⁻¹. The calculated

TABLE II

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR WHITE MoO₃·H₂O[a]

l	Fo	Fc
0,0,L		
2	485	515
3	336	350
4	267	272
5	196	197
0,1,L		
-5	165	235
-4	331	371
-3	292	300
-2	106*	124
2	349	373
3	346	350
4	227	294
0,2,L		
-5	229	285
-4	419	428
-3	485	459
-2	771	683
1	406	376
2	238	199
3	188	175
0,3,L		
-4	203	139
-2	79*	88
-1	324	207
0	258	182
1	378	330
1,-3,L		
-1	192	107
0	389	303
1	442	398
3	422	295
4	410	349
1,-2,L		
-3	373	414
-2	637	598
-1	602	536
0	529	400
1	490	406
2	146*	111
3	56*	58
4	130*	181
5	145*	224
1,-1,L		
-5	71*	31
-4	23*	112
-3	6*	27
-2	259	253
-1	527	508
3	418	429
4	410	369
5	275	294
1,0,L		
-5	366	402
-4	452	493
-3	759	813
-2	518	570
2	7*	3
3	312	330
4	264	314
5	296	345
1,1,L		
-6	41*	36
-5	136*	168
-3	373	385
-2	503	502
2	399	409
3	309	325
4	217	249
1,2,L		
-6	296	351
-5	426	448
-4	598	583
-3	289	294
-2	250	220
-1	8*	25
0	8*	50
1	152*	147
2	365	360
3	315	336
1,3,L		
-4	285	332
-3	301	353
0	316	265
2,-3,L		
-3	185	164
-2	146*	146
-1	215	199
0	413	301
1	555	417
2	487	395
3	445	392
4	578	555
2,-1,L		
-4	409	450
-3	536	573
-2	594	625
0	359	358
1	270	268
2	110*	117
3	114*	23
4	210	221
2,0,L		
-6	221	242
-3	185	39
-2	405	419
0	657	676
1	443	464
2	517	551
3	622	636
4	375	402
2,1,L		
-6	318	342
-5	407	442
-4	579	600
-3	495	498
-1	272	284
0	102*	146
2	169	150
3	250	280
2,2,L		
-6	10*	19
-5	9*	9
-4	154*	138
-3	386	352
-2	529	440
-1	689	596
0	467	437
1	507	505
2	476	464
2,3,L		
-4	260	275
-3	203	129
-1	117*	34
3,-3,L		
-1	68*	63
1	152*	146
2	345	267
3,-2,L		
-3	362	391
-2	458	445
-1	432	440
0	467	411
1	719	614
2	472	396
3	224	237
3,-1,L		
-5	260	302
-4	155	153
-3	83*	120
-2	24*	100
-1	332	321
0	279	276
1	451	455
2	373	399
3	457	503
4	365	405
3,0,L		
-6	108*	138
-5	371	412
-4	470	492
-3	527	588
-2	351	426
-1	446	505
0	768	757
1	373	411
2	214	199
3,1,L		
-6	184	213
-5	9*	33
-4	39*	46
-3	194	221
-2	341	332
-1	402	405
0	507	511
1	389	424
2	460	490
3	326	334
3,2,L		
-6	376	388
-5	438	437
-4	501	512
-3	349	341
-2	477	438
-1	531	468
0	264	242
1	51*	73
3,3,L		
-4	233	205
-3	348	268
-2	459	387
4,-2,L		
-3	223	243
-2	230	298
-1	129*	131
1	37*	35
2	230	206
3	218	-224
4,-1,L		
-4	421	434
-3	380	385
-2	472	494
-1	475	499
0	650	660
1	500	529
2	154*	201
3	110*	133
4,0,L		
-6	269	322
-5	253	273
-3	325	316
0	184	195
1	300	294
2	270	297
3	313	330
4,1,L		
-6	275	319
-5	419	447
-4	426	432
-2	467	464
-1	609	625
0	373	391
1	111*	130
2	10*	45
4,2,L		
-5	176	188
-4	183	176
-3	92*	37
-2	67*	112
-1	254	277
0	263	277
5,-2,L		
-1	336	360
0	406	396
1	450	445
5,-1,L		
-4	298	348
-3	371	394
-2	307	333
-1	89*	55
0	9*	8
1	209	165
2	152*	192
5,0,L		
-5	178	197
-4	140*	185
-3	312	264
-2	407	437
-1	440	474
1	475	491
1	297	260
2	208	227
5,1,L		
-5	265	293
-4	341	360
-3	215	192
-2	3*	53
-1	135*	127
3	193	238
1	195	219
5,2,L		
-5	260	223
-4	313	318
-3	369	366
-2	398	408
-1	369	387
6,-1,L		
-4	122*	90
-3	142*	136
-2	320	339
-1	404	430
0	394	412
6,0,L		
-5	327	344
-4	331	352
-3	333	361
-2	108*	141
-1	62*	75
0	88*	73
6,1,L		
-5	138*	175
-4	214	213
-3	396	398
-2	397	421
-1	379	430
7,0,L		
-3	168	135
-2	247	248

[a] The columns are l, F_o, F_c. Unobserved reflections are those less than 3 $\sigma(I)$ and are marked by *.

absorption factors ranged from 1.063 to 1.106. The intensities were reduced to F_0 in the usual way by applying Lorentz and polarization factors.

All the computations, including subsequent refinement of the structure, were performed using the program system XRAY (7). The positions of the Mo atoms could readily be deduced from the peak location in a three-dimensional Patterson map. The resulting Mo–Mo distances of about 3.40 and 3.70 Å seemed to be reasonable values corresponding to MoO_6-octahedra connected by common edges or corners found in related compounds as MoO_3 (6) and $(NH_4)_6Mo_8O_{27} \cdot 4H_2O$ (8).

Structure factor calculations based on refined positions of these Mo atoms yielded a conventional R index of 0.24

$$R = \sum (||F_0| - |F_c||)/\sum (|F_0|).$$

The scattering factors used were those for the neutral atoms tabulated in International Tables for X-Ray Crystallography (9). A difference electron density map showed the positions of four oxygen atoms in the centric space group $P\bar{1}$ and of eight oxygen atoms in the acentric $P1$. Although the distribution of the intensities in a preliminary calculated Wilson plot showed acentric behaviour, all attempts of a refinement in $P1$ resulted in oscillations of the shifts in least-squares refinement and extremely large standard deviations of the calculated bond lengths. The subsequent refinement in the centric space group $P\bar{1}$ was more successful and was performed to a R-index of 0.14 with isotropic temperature factors. At this stage of the refinement 39 reflexions which were affected by uncertainties in the intensity measurements, e.g., by pronounced asymmetry of the background measurements or by incompletely corrected absorption effects were excluded from further calculations. Anisotropic least-squares refinement with unit weights for all reflexions decreased R to the final value of 0.088 including all of the remaining 204 observed reflexions. However, since aniso-tropic temperature factors shifted to negative values in this least-squares refinement and had to be set positive definite by the program, we do not assign physical meaning to the

calculated anisotropic temperature factors but consider it as a consequence of the aniso-tropic disorder in the crystal described earlier.

The final atomic positional coordinates are listed in Table I. Observed and calculated structure factors are presented in Table II.

Description of the Structure

Figures 5 and 6 show projections of the crystal structure of white $MoO_3 \cdot H_2O$ parallel to [001] respectively to [010]. The resemblance between the idealized prediction from Figs. 3a and 3b and the structure actually deter-mined is evident.

The dominating element is an infinite double chain of edge-linked $[MoO_5(H_2O)]$-octahedra parallel to [010]. Each octahedron has two common edges with neighboured ones. In addition to the three bridge-forming oxygen atoms, each molybdenum atom is coordinated
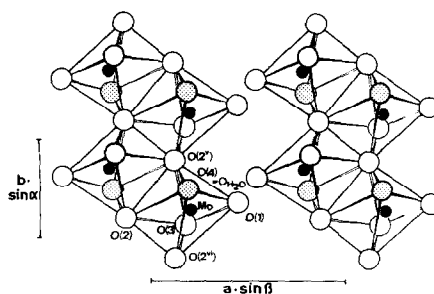

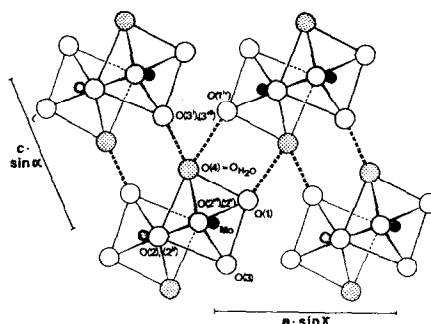FIG. 5. Projection of the crystal structure of white molybdenum trioxide–monohydrate parallel to [001].

FIG. 6. Projection of the crystal structure of white molybdenum trioxide–monohydrate parallel to [010]. Probable hydrogen bonds are indicated by dotted lines.

TABLE III

Interatomic Distances and Bond Angles in White $MoO_3 \cdot H_2O$

(a) *Mo–O octahedron:*

Mo–O(1)	1.66(3) Å	O(1)–Mo–O(2^{vi})	104.3
Mo–O(2)	2.28(3)	O(2)–Mo–O(3)	95.6
Mo–O(3)	1.65(4)	O(2)–Mo–O(4)	76.9
Mo–O(4)	2.37(4)	O(2)–Mo–O(2^{v})	75.1
Mo–O(2^{v})	1.98(3)	O(2)–Mo–O(2^{vi})	70.7
Mo–O(2^{vi})	1.91(3)	O(3)–Mo–O(4)	172.0
		O(3)–Mo–O(2^{v})	96.8
O(1)–Mo–O(2)	161.0° ^{b}	O(3)–Mo–O(2^{vi})	98.3
O(1)–Mo–O(3)	103.3	O(4)–Mo–O(2^{v})	78.9
O(1)–Mo–O(4)	84.3	O(4)–Mo–O(2^{vi})	81.9
O(1)–Mo–O(2^{v})	104.1	O(2^{v})–Mo–O(2^{vi})	143.7

(b) *Mo–Mo distances:*

Mo–Mo$^{(ii)}$	3.700(5) Å	Corner sharing octahedra
Mo–Mo$^{(iii)}$	3.700(5)	
Mo–Mo$^{(v)}$	3.383(5)	Edge sharing octahedra
Mo–Mo$^{(vi)}$	3.425(5)	

(c) *Short O–O distances between neighboured double chains, corresponding to possible hydrogen bonds:*

O(4)–O(1^{iv})	2.76(4) Å
O(4)–O(3^{i})	2.85(4)
O(4)–O(3^{vii})	3.14(5)

^{a} Standard deviations are given in parenthesis. The superscripts indicate the transformations applied to the x, y, z values given in Table I: (i) $x, 1 + y, 1 + z$, (ii) $x, 1 + y, z$, (iii) $x, -1 + y, z$, (iv) $1 - x, -y, 1 - z$, (v) $-x, -y, -z$, (vi) $-x, -1 - y, -z$, (vii) $x, y, 1 + z$.

^{b} $\sigma_{(O-Mo-O)} = 1.1{-}1.7°$.

by two terminal oxygen atoms and one terminal water molecule. Although the hydrogen atoms could not be directly localized, it becomes evident from the interatomic distances that O(4) has to be the water oxygen atom.

The octahedral double chains are linked among each other through possible hydrogen bonds only. This fact can explain the characteristic needle shape of the crystals and the marked tendency to disorder by a more or less pronounced tilt of subneedles around [010].

Table III contains the most important interatomic distances and angles. The [MoO$_5$-(H$_2$O)]-octahedron exhibits a strong characteristic distortion, containing two very short Mo–O bonds of 1.66 and 1.65 Å to the terminal oxygens O(1) and O(3), two medium Mo–O bonds of 1.98 and 1.91 Å to the bridge-

forming oxygens O(2^{v}) and O(2^{vi}), and finally two very long Mo–O distances of 2.28 and 2.37 Å to the third bridging oxygen atom O(2) and the water oxygen O(4). The average of the six Mo–O distances is 1.975 Å and lies very close to the respective value for MoO$_3$ of 1.981 Å (6).

The strongly bonded terminal oxygen atoms O(1) and O(3) stand in *cis* position to each other, whereas the longest distance to a bridge-forming oxygen O(2) and the bond to the water molecule O(4) are located in *trans* position relative to O(1) and O(3). This kind of octahedral distortion with two very short *cis*-Mo–O bonds, two very long bonds in *trans* position to them and two medium distances to the remaining oxygen atoms seems quite common in molybdenum compounds with oxygen and has been found, e.g.,

in MoO_3 (5, 6) and in $Na_2Mo_2O_7$ (10) as well. The Mo–O bonds in the order of 1.66 Å to the terminal oxygens belong to the shortest ones which were ever found in molybdenum-oxygen compounds and point to a considerable amount of π-bonding character.

If the two ligands with the longest Mo–O distances are neglected, the structure may be alternatively described as built up from chains [010] of corner-linked distorted MoO_4-tetrahedra and isolated water molecules. In a very similar way, Kihlborg (6) has characterized the structure of MoO_3 as a transitional state between octahedral and tetrahedral coordination.

Evidence for hydrogen bonds between the octahedral double chains follows from the O–O distances around the water oxygen O(4) in Table III. Relatively short distances from O(4) to oxygen atoms of neighboured octahedral double chains are 2.76 Å to $O(1^{iv})$, 2.85 Å to $O(3^i)$, and 3.14 Å to $O(3^{vii})$. These values are suggesting medium strength, respectively, weak hydrogen bonding between otherwise isolated chains. If one of the water hydrogen atoms is assumed definitely bonded to $O(1^{iv})$, it remains open whether one of the two other contacts represents the second water hydrogen bridge. If both of them should be involved in hydrogen bond formation, a statistical distribution of the second water hydrogen atoms over both possibilities had to be adopted. In Fig. 6, the most probable hydrogen bonds are indicated by dotted lines.

Conclusions

From the comparison between the structure proposal for white $MoO_3 \cdot H_2O$ derived by means of topotactical considerations and its independently determined actual crystal structure follows that the study of topotaxy can represent a powerful tool in predicting structures. In the present case, a so far unknown educt crystal decomposes by loss of one water molecule per formula unit into a perfect pseudomorph built up from highly oriented crystallites of a known product structure. There happens nothing else than an escape of the O(4) atoms as water, a subsequent

13

linking of the octahedral double chains over common corners O(3) to form layers and finally a shift of the triclinic angles α, β, γ to the 90° angles of the orthorhombic MoO_3 structure.

One could of course imagine also the reverse case, where an educt crystal of known structure is topotactically transformed into a highly oriented, but structurally unknown product, and conclusions about the product structure may be drawn. In between there is the case treated by Günter (1): An educt $MoO_3 \cdot 2H_2O$ is transformed into a product MoO_3, both of known structure, with the yellow form of $MoO_3 \cdot H_2O$ acting as a structurally unknown intermediate. Although the proposal for its structure derived from topotaxy has not yet been verified independently, the result of this work is well suited to support it.

Even when all of the structures involved in a chemical reaction are already known, the study of topotactical phenomena can be of considerable interest, enhancing the understanding for structural relationships and enabling conclusions regarding reaction mechanisms. Specially well suited for such studies are all structures with pronounced elements like chains or layers. They are by no means restricted to thermal decompositions, but can be undertaken in solid–liquid, solid–gas, and solid–solid reactions as well.

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