Preparation and Probable Structure of Layered Complexes of Vanadyl Phosphate with 1-Alkanols and 1,ω-Alkanediols

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The direct reaction of liquid (or melted solid) 1-alkanols or 1,ω-alkanediols with solid finely ground $VOPO_4\cdot 2H_2O$ in a microwave field leads to layered complexes of the composition $VOPO_4\cdot 2C_nH_{2n+1}OH$ ($n = 1-18$) or $VOPO_4\cdot C_nH_{2n}(OH)_2$ ($n = 2-10$), respectively. The results of X-ray diffraction analysis show that the structures of all of these polycrystalline complexes (intercalates) retain the original layers of $(VOPO_4)_\infty$. The molecules of alcohols are placed between the host layers in a bimolecular way, being anchored to them by donor−acceptor bonds between the oxygen atom of an OH group and a vanadium atom as well as by hydrogen bonds. The molecules of diols, on the other hand, using similar bonds, form, in monomolecular arrangement, bridges linking the adjacent layers of the host. The aliphatic chains of both intercalated alcohol and diol molecules possess *all-trans* configurations, and their axes are perpendicular to the host layers.

Introduction

A number of recently published papers have reported attempts to find the structural principles governing the intercalation of aliphatic molecules (with terminal functional groups) between the layers of hosts. With this aim have been investigated the intercalations of, e.g., aliphatic alcohols, diols, amines with unbranched carbon chains into some natural silicates,[1−4] vanadyl or niobyl sulfates and phosphates,[5−7] vanadyl alkylphosphonates,[8] and other similar hosts.

Recently we determined the composition and lattice parameters of layered complexes formed by intercalation of aliphatic alcohols (C_1-C_8),[9,10] amines (C_2-C_{10}),[11] and carboxylic acids (C_1-C_4)[12] into three isostructural layered hosts: vanadyl phosphate (V^VOPO_4), vanadyl arsenate (V^VOAsO_4), and vanadyl sulfate ($V^{IV}OSO_4$).

From the experiments carried out so far it follows that the intercalated molecules of the given kind are always anchored to the host layers by their functional groups.[13,14] At room temperature and with sufficient amounts of the host molecules, the guest chains in the complex possess the *all-trans* configuration[1] and their axes form with the host layers either an acute angle (usually 55−60°)[11,13] or a right angle,[1,8,13] the guest layers being most often bimolecular[8,9,10,13] but also sometimes monomolecular.[8,12]

So far it has not been possible to apply direct X-ray diffraction analysis to structure determination of the complexes mentioned because these substances show no tendency to form crystals of sufficient magnitude and suitable mechanical properties (they have a gelatinous consistency) and all of them are readily hydrolyzed in air.

Using a new preparation method, we have now succeeded in obtaining the $VOPO_4$ complexes with an extensive homologous series of 1-alkanols up to 1-octadecanol, i.e., also those that are solids at room temperature. Similarly we have prepared the $VOPO_4$ complexes with 1,ω-alkanediols up to 1,10-decanediol. Having determined the lattice parameters of all of these complexes, we suggest the most probable arrangement of the alcohol and diol molecules between the host layers.

Experimental Section

Preparation. Vanadyl phosphate dihydrate $VOPO_4\cdot 2H_2O$ (for structure see refs 15 and 16) was obtained by long-term boiling of a V_2O_5 suspension in aqueous phosphoric acid.[17]

The layered complexes with alcohols and diols were prepared by suspending microcrystalline $VOPO_4\cdot 2H_2O$ (ca. 1 g; grain size 0.01−0.08 mm) in dry liquid alcohol (ca. 7−10 g) or mixing with solid alcohol, and subsequent short exposure (0.5−5 min) to a microwave field; the reaction mixture was placed in a 15 cm^3 glass flask equipped

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with a reflux condenser and put into the waveguide of microwave generator with stirring and heating. After cooling, the solid product formed was filtered off. When the starting material was a solid alcohol, the suspension was separated from the melt by hot filtration. The samples for X-ray diffraction analyses were left wetted with a small residue of the respective alcohol, and also in the preparations starting from melted alcohols the residues of solid alcohols were not removed. On the other hand, the samples for thermogravimetric and differential thermal analyses and for IR spectral measurements were washed with pentane (intercalates with liquid alcohols) or toluene followed by pentane (intercalates with solid alcohols).

The microwave apparatus used for preparing the complexes was specially constructed by Radan Ltd., Pardubice, the Czech Republic. It operates at a frequency of 2450 ± 30 MHz with a total generator output of 800 W, out of which 30−50 W is absorbed by the reaction mixture. The metal waveguide with a square cavity (5.2 × 5.2 cm) is equipped with an opening of 2 cm diameter, which serves for locating the reaction vessel into the waveguide axis.

Analyses. The TGAs of the intercalates were performed with a Derivatograph MOM (Hungary), the measurements being carried out in the temperature interval of 30−700 °C in an N_2 atmosphere at heating rates of 5 °C min^{-1}. The weight of the samples was 100 mg.

In some cases, particularly with complexes of solid alcohols, the composition was determined by elemental analysis (C, H).

Diffraction Measurements. The powder data of the intercalates with a minor surplus of the guest alcohol were obtained with an X-ray diffractometer (HZG-4, Germany) using $CuK\alpha_1$ radiation ($\lambda = 1.5405$ Å) with discrimination of the Cu $K\beta$ by an Ni filter. The $CuK\alpha_2$ intensities were removed from the original data. Silicium ($a = 5.430\ 55$ Å) was used as internal standard. Diffraction angles were measured from 1.5° to 50° (2Θ). The obtained data were refined by a least-squares program minimizing $(2\Theta_{exp} - 2\Theta_{calc})^2$.

IR Spectral Measurements. The absorption IR spectra of all of the complexes in Nujol suspension were recorded in the region 4000−400 cm^{-1} on a Perkin-Elmer 684 spectrometer.

Calculation of Steric Demands of 1-Alkanol and 1,*ω*-Alkanediol Molecules. Introduction of foreign molecules between host layers such as layers of vanadyl phosphate is usually accompanied by a distinct increase in the basal spacing *c* of the solid. The magnitude of this change is governed by the van der Waals dimensions of the entering molecules, their numbers, their way of location in the interlayer space, and also the lengths of the bonds anchoring these molecules to the host layers.

The 1-alkanol and 1,*ω*-alkanediol molecules, provided that their chains are present in the *all-trans* configuration, can roughly be viewed as rotational cylinders with the van der Waals diameters of about 5.20 Å. If the bases of the cylinders are located (see Figure 1) in such a way that one of them crosses the oxygen atom of the OH group and the other the carbon atom of the CH_3 group (in 1-alkanols) or both cross the oxygen atoms of terminal OH groups (in 1,*ω*-alkanediols), then the heights of these cylinders are expressed by the following equations:

$$h_{alc} = (n - 1)l_{CC} \sin \beta + l_{OC} \sin \gamma \tag{1}$$

$$h_{diol} = (n - 1)l_{CC} \sin \beta + 2l_{OC} \sin \gamma \tag{2}$$

where *n* represents the number of carbon atoms in the unbranched aliphatic chain of the molecule, *l* designates bond length ($l_{CC} = 1.537$ Å, $l_{OC} = 1.40$ Å),[18] and β and γ are the angles characterizing the spatial arrangement of the aliphatic chain ($^1/_2 \angle CCC = \beta = 56.3°$, $\angle OCC - \beta = \gamma = 53.2°$).[19]

The demands of the guest 1-alkanol molecule for the gapping of host layers are obviously given (see Figure 2a) by the sum of the perpendicular distance (*b*) of the oxygen atom of the OH group from the host layer, the chain length (h_{alc}), and the van der Waals radius of the methyl group (r_{CH3}). The contribution of the length of the molecule is, of course, affected by the angle (α) formed by the axis of its chain and the host layer.

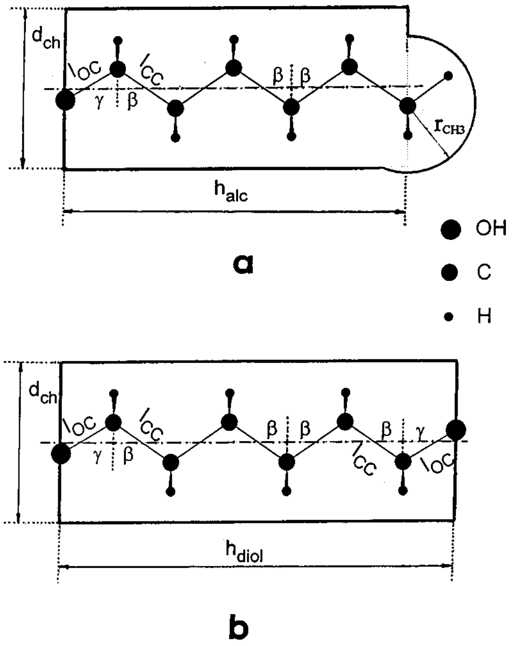

Figure 1. Representation of simplified shape and dimensions of molecules of 1-alkanols (a) and 1,*ω*-diols (b). All of the aliphatic chains assume *all-trans* configurations.

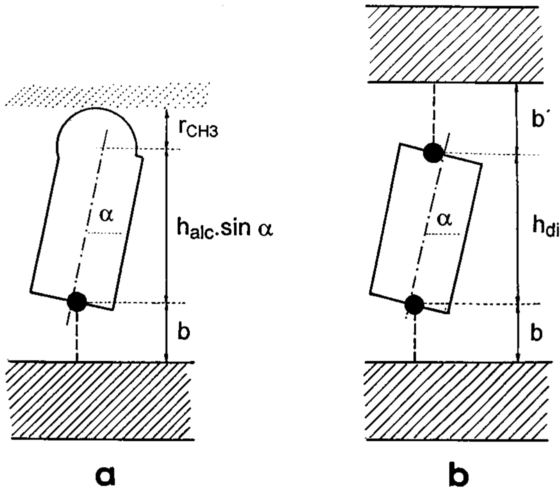

Figure 2. The basic idea about steric demands of 1-alkanol (a) and 1,*ω*-diol (b) molecules for necessary gapping of rigid layers of host.

Similarly, the steric demands of 1,*ω*-alkanediol molecules (Figure 2b) are given by the perpendicular distances oxygen−layer (*b*, *b′*) which need not be the same, by the length of the molecule (h_{diol}), and by the inclination of its axis (α).

The transition from considering the steric demands of a single molecule to considering those of whole layers of such molecules can bring certain complications. For instance, the guest particles can form not only a monomolecular layer as that expected with diols (Figure 3a) but also a bimolecular layer (Figure 3b), which is more likely in the case of monobasic alcohols. In the latter case the terminal methyl groups must slightly “overlap”, and the demand of the bimolecular layer for gapping of the host structure must thereby be decreased (−*k*). In the case of diols it cannot be excluded that their molecules could be anchored by both OH groups to the same host layer, which would result in the basal spacing of such complexes not increasing with increasing length of the aliphatic chain.

In spite of these and other possible complications, it is obvious that there is a simple way of deriving the probable geometry of incorporation of the chains from the experimentally determined dependence of basal spacing (*c*) of the complexes prepared on the number of carbon atoms

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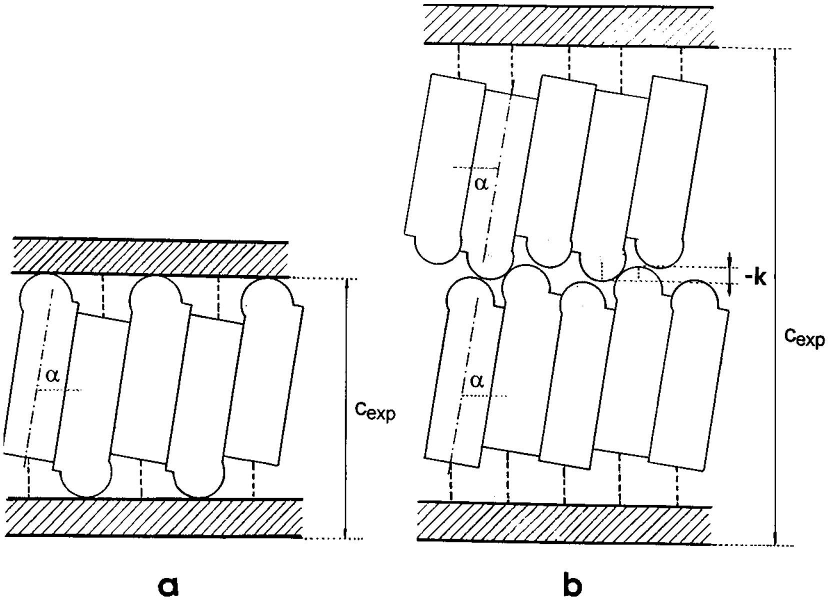

Figure 3. Steric demand of monomolecular (a) and bimolecular (b) layers of 1-alkanol in interlayer space of host.

(n) in the chain of intercalated molecules. In most cases the basal spacing c will change with n according to the relation

$$\Delta c/\Delta n = m(\Delta h/\Delta n)\sin\alpha \qquad (3)$$

where m is the number of layers of guest molecules in one interlayer space of the host, α is the inclination of the axis of the carbon chain of the individual molecule, and the ratio $\Delta h/\Delta n$ (calculated by differentiating eq 1 or 2) has the value of 1.279 for both 1-alkanols and 1,ω-alkanediols.

Results and Discussion

Preparation and Composition of Complexes. The application of microwave radiation to the suspension of hydrated host ($VOPO_4{\cdot}2H_2O$) in an excess of liquid guest has been described recently.[20] It turned out to be a highly efficient method of preparation of the respective intercalates in the case of application of liquid or melted 1-alkanols or 1,ω-alkanediols. The absorption of microwaves by the water molecules present in the structure of the hydrate results in rapid removal of these molecules from the space between the $(VOPO_4)_\infty$ to the surrounding dry alcohol and in exfoliation of host.

During exposure to a microwave field, the reaction mixture with volatile alcohol was heated to its boiling point. If the alcohol intercalated had a longer chain, then the temperature of the reaction mixture reached ca. 150 °C. The alcohols of higher molecular weight required a longer exposure and, hence, also a higher temperature. After finishing the exposure and partially cooling the reaction mixture the $(VOPO_4)_\infty$ are reassociated taking the molecules of new guest between them a microcrystalline intercalate without water is formed. In this way the following exchange takes place:

$$VOPO_4{\cdot}2H_2O(s) + xG(l) \rightarrow VOPO_4{\cdot}xG(s) + 2H_2O(l,g)$$

The samples of intercalates prepared in the given way were crystalline, and their diffractograms showed a series of relatively sharp (00l) reflections (see Table 1). The a parameter of the tetragonal lattice of the intercalates was determined from the position of diffraction line (200), which had a relatively low intensity in all of the samples due to formation of the preferred orientation. The (hk0) and (hkl) lines were not found in most intercalates. The values found for the lattice parameters a and c are presented in Table 1 for all the intercalates prepared.

Table 1. Lattice Parameters a, Number of (00l) Lines, and Measured and Calculated Basal Spacings of the Intercalates of Vanadyl Phosphate with 1-Alkanols and 1,ω-Alkanediols

composition	a (Å)	no. of lines (00l)	basal spacing (Å)	
			measd	calcd
$VOPO_4{\cdot}2CH_3CH_2OH$	6.22	5	13.17	12.26
$VOPO_4{\cdot}2CH_3(CH_2)_2OH$	6.19	4	14.36	15.04
$VOPO_4{\cdot}2CH_3(CH_2)_3OH$	6.21	3	17.90	17.42
$VOPO_4{\cdot}2CH_3(CH_2)_4OH$	6.21	4	19.71	20.12
$VOPO_4{\cdot}2CH_3(CH_2)_5OH$	6.22	5	22.73	22.58
$VOPO_4{\cdot}2CH_3(CH_2)_6OH$	6.22	6	25.09	25.26
$VOPO_4{\cdot}2CH_3(CH_2)_7OH$	6.21	5	28.44	27.76
$VOPO_4{\cdot}2CH_3(CH_2)_8OH$	6.19	7	30.62	30.40
$VOPO_4{\cdot}2CH_3(CH_2)_9OH$	6.19	9	33.50	32.92
$VOPO_4{\cdot}2CH_3(CH_2)_{10}OH$	6.20	4	35.68	35.54
$VOPO_4{\cdot}2CH_3(CH_2)_{11}OH$	6.20	8	38.24	38.08
$VOPO_4{\cdot}2CH_3(CH_2)_{12}OH$	6.21	4	41.07	40.70
$VOPO_4{\cdot}2CH_3(CH_2)_{13}OH$	6.21	7	43.40	43.24
$VOPO_4{\cdot}2CH_3(CH_2)_{14}OH$	6.21	6	46.21	45.86
$VOPO_4{\cdot}2CH_3(CH_2)_{15}OH$	6.19	5	48.36	48.40
$VOPO_4{\cdot}2CH_3(CH_2)_{16}OH$	6.21	5	50.92	51.02
$VOPO_4{\cdot}2CH_3(CH_2)_{17}OH$	6.21	5	54.01	53.58
$VOPO_4{\cdot}HO(CH_2)_2OH$		2	8.56	8.82
$VOPO_4{\cdot}HO(CH_2)_3OH$		1	10.05	10.01
$VOPO_4{\cdot}HO(CH_2)_4OH$	6.19	3	11.54	11.36
$VOPO_4{\cdot}HO(CH_2)_5OH$	6.22	3	12.40	12.59
$VOPO_4{\cdot}HO(CH_2)_6OH$	6.20	4	14.04	13.92
$VOPO_4{\cdot}HO(CH_2)_7OH$	6.20	4	15.13	15.17
$VOPO_4{\cdot}HO(CH_2)_8OH$	6.21	4	16.68	16.50
$VOPO_4{\cdot}HO(CH_2)_9OH$	6.20	5	17.92	17.76
$VOPO_4{\cdot}HO(CH_2)_{10}OH$	6.20	3	18.95	19.07

The results of thermogravimetry of all the complexes together with several checking analyses of carbon and hydrogen content showed that the intercalates of 1-alkanols (except for methanol) have the stoichiometric quotient $x = 1.92 \pm 0.07$, hence their composition most probably corresponds to the formula $VOPO_4{\cdot}2C_nH_{2n+1}OH$. In contrast, 1,$\omega$-alkanediols form complexes with $x = 0.95 \pm 0.08$ corresponding to the formula $VOPO_4{\cdot}C_nH_{2n}(OH)_2$.

According to our preliminary results, vanadyl phosphate dihydrate reacts similarly also with alkylamines, alkanoic acids, amino acids, and some other substances. It is worth mentioning that no reaction in the microwave field takes place if anhydrous

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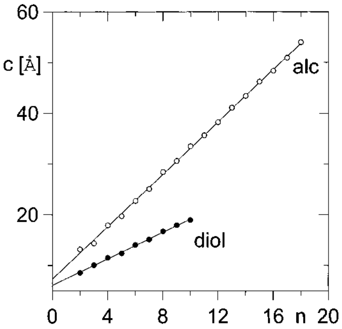

Figure 4. The dependence of basal spacing of all prepared complexes on the number of carbon atoms in the aliphatic chain of intercalated alcohols and diols.

$VOPO_4$ instead of the dihydrate is suspended in any of the above-mentioned liquid guests.

Orientation of Chains with Respect to Host Layers. Figure 4 presents graphically the dependence of basal spacing *c* on the number of chain carbon atoms in the intercalated molecules.

For the intercalates of 1-alkanols the set of experimental points fits the straight line $c = (2.58 \pm 0.02)n + (7.33 \pm 0.2)$ with the correlation coefficient $r = 0.9996$. The slope found for this straight line corresponds very well (see eq 3) to the situation of bimolecular arrangement of the guest 1-alkanol in the interlayer space ($m\Delta h_{alc}/\Delta n = 2.56$, $m = 2$) and perpendicular orientation of the chain axes with respect to the host layers ($\alpha = 90°$).

The layered complexes of 1,*ω*-alkanediols again show a linear dependence of their *c* parameters on the number of carbon atoms. The fitting straight line has the equation $c = (1.30 \pm 0.02)n + (6.10 \pm 0.12)$ ($r = 0.9990$). Its slope is half of that in the previous case and indicates a monomolecular arrangement of diol molecules ($m\Delta h_{diol}/\Delta n = 1.30$, $m = 1$), but again with the chains perpendicular to host layers ($\alpha = 90°$).

This conclusion perfectly agrees with the difference found between the stoichiometries of the intercalates: it is 1:2 for 1-alkanols and 1:1 for 1,*ω*-alkanediols.

IR Spectral Analysis. The above idea about the structure of the intercalates is supported by analysis of their IR spectra. The valence vibration band of the vanadyl group $\nu(V{=}O)$ in vanadyl salts was usually found within the frequency region 1050−980 cm^{-1} (see ref 21), and it is sensitive to the donor ability of the ligand which coordinates the vanadium atom at the position opposite to the oxygen atom.[22] In anhydrous vanadyl phosphate $\nu(V{=}O)$ is found at 1027 cm^{-1}. In this compound the above-mentioned position is occupied by an oxygen atom from the neighboring layer, but with respect to the large bonding distance between both of these atoms (2.50 Å),[15] it can rather be considered unoccupied. In the hydrate $VOPO_4 \cdot 2H_2O$, where water molecules are bound to vanadium atoms,[23] the wavenumber of the band mentioned is 1015 cm^{-1}.

The IR spectra of all of the intercalates of alcohols and diols contain the $\nu(V{=}O)$ band in the region 1010−1020 cm^{-1}, overlapping the intense bands of the PO_4 tetrahedron. This indicates the presence of a donor−acceptor bond between an oxygen atom of the alcohol or diol and the vanadium atom analogous to the bond of water molecules to the $(VOPO_4)_\infty$ layer in the structure $VOPO_4 \cdot 2H_2O$.[25]

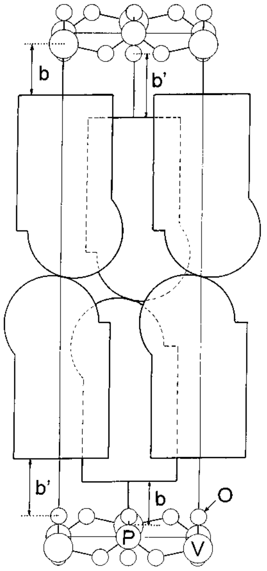

Figure 5. The principles of location of 1-alkanol molecules in the interlayer space of the $VOPO_4$ host. The donor−acceptor bond length $O_{alc} \rightarrow V$ is denoted as *b*. The hydrogen bond length $O_{alc}-H\cdots O_{V=O}$ is denoted as *b′*. The axial heights of the cylinders representing the aliphatic chains are slightly shortened.

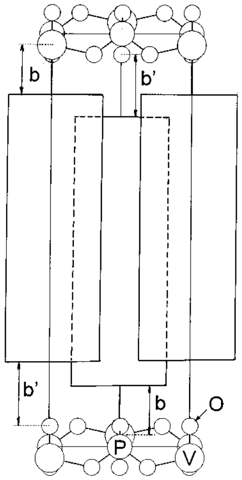

Figure 6. The principles of location of 1,*ω*-alkanediol molecules in the interlayer space of the $VOPO_4$ host. The diol molecules are anchored to the layers by the same bonds as in the case of 1-alkanols.

Weak and medium intense bands were found in the regions 3560−3530 cm^{-1} and 3400−3300 cm^{-1}. The first of these regions can be assigned to the mutual interaction of these alcohols,[24] and the second could possibly correspond to the valence vibration of an OH group affected by the interaction between the alcohol molecule and the host. Broad intense bands in the region 3180−3165 cm^{-1} belong most probably to the interaction of the alcohol with the host lattice resulting in the hydrogen bonding between oxygen atoms of the host lattice and the OH group of the alcohol molecules.

Probable Structure of Intercalates. If we take into account both the stoichiometry of the complexes prepared and the fact that the aliphatic chains of the guest molecules have their axes perpendicular to the host layers, and finally also the fact that probably half of the OH groups of the intercalated molecules are bound by a donor−acceptor bond to vanadium atoms while

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the other half are anchored to oxygen atoms of vanadyl groups through a hydrogen bridge, we can suggest a possible structure of these compounds: it is schematically represented in Figures 5 and 6.

If all of the experimentally found values of basal spacing of complexes with diols are treated in such a way that the calculated chain lengths (h_{diol}) as well as the coordinates of V and O atoms in the $(\mathrm{VOPO_4})_\infty$ layer are subtracted from them, we get a remarkably constant and well-acceptable value of $(b + b') = 3.86 \pm 0.15$ Å in all of the complexes (b is the bond length between the diol oxygen atom and vanadium, and b' is the bond length in the hydrogen bridge of $\mathrm{O_{diol}{-}H{\cdots}O_{V{=}O}}$). Reintroducing this mean value of distance $(b + b')$, we obtain the c parameter for all of the complexes with diols (see the c_{calc} values given in Table 1).

If the bond lengths b and b' are retained also in the complexes with 1-alkanols (which is highly probable), a simple calculation will show that the "overlap" of methyl groups $(-k)$ in the respective bimolecular layers must amount to about -2.60 ± 0.15 Å. The results of back-calculated c_{calc} values of the complexes with 1-alkanols are given in Table 1.

It is useful to evaluate the steric demands of a set of aliphatic chains covering a certain plane and oriented perpendicularly to it. The projection of a chain in its *all-trans* conformation on the plane perpendicular to its axis represents an ellipse of 5.2 × 4.2 Å dimensions. Figure 7 gives an example of a possible way of covering a $(\mathrm{VOPO_4})_\infty$ layer with the alcohol molecules.

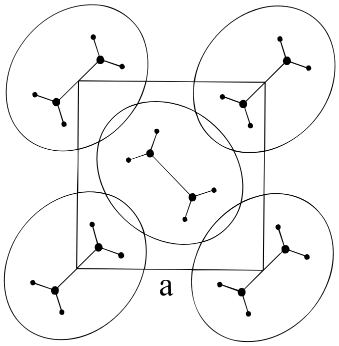

Figure 7. Covering of a host layer by perpendicularly oriented aliphatic chains of the intercalated molecules. The van der Waals diameter of chains is represented by an ellipse of 5.2 × 4.2 Å dimensions.

In spite of the highly satisfactory agreement between the results of the calculations carried out, our balance approach must only be considered a mere attempt at speculative finding of the structural principles governing the location of alcohol and diol molecules between the layers of one of the hosts.

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