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DEFORMATIONS OF THE SOLID PHASE OF DISPERSED SYSTEMS

UNDER THE INFLUENCE OF ADSORBED WATER AND ORGANIC COMPOUNDS

P. P. Olodovskii and L. A. Malkova   UDC 541.182

Diffractometric data are used for determining the variation of the volume of the crystal lattice of kaolinite under the simultaneous adsorption of water and organic compounds.

The effect of adsorbed water alone on the variation of the parameters and volume of the crystal lattice of kaolinite was studied in [1]. In what follows, we present the results of further investigations. The techniques and methods of measurement remained essentially the same. We used the method of x-ray diffractometry with a DRON-2.0. All the experimental results are shown in Fig. 1, from which it can be seen that the volume of an elementary cell of kaolinite expands during the hydration process. It should be noted that this expansion is determined by the structure of the organic-compound molecules. This phenomenon can be explained by the following.

Calculation of the Deformations of the Crystal Lattice of Kaolinite. On the basis of [2], for deformations which vary only slightly within the limits of some constants of the lattice, the change in volume can be represented as

$$\frac{\Delta V}{V} = \frac{\Delta E_F}{E_F},\qquad(1)$$

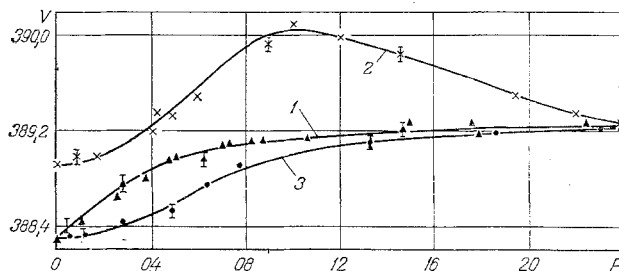where E_F is the Fermi energy of the crystal lattice.

Fig. 1. Variation of the volume of an elementary cell of the natural form of Glukhovetsk kaolinite adsorbing: 1) water; 2) water and toluene; 3) water and nitrobenzene. V in $\overset{\circ}{A}^3$; P in %.

According to the electronic theory of catalysis [3], almost all the main adsorption and catalytic properties of the surface of the crystal lattice of the adsorbent are determined by the position of the Fermi level. The very fact that there exists a "strong" form of chemisorption, i.e., a form in which a bound particle holds a free electron or a free hole on itself (or near itself), implies a charging of the semiconductor — the formation in the layer near the surface of a volumetric charge which has a sign opposite to that of the surface charge and compensates it, leading to distortion of the energy zones near the surface of the crystal.

Thus, the position of the Fermi level on the surface of the crystal, in comparison with its position in the interior, is found to be shifted by an amount $\Delta E_F = E^{+}_{F(V)} - E^{+}_{F(S)} = E^{-}_{F}(S) \to E^{-}_{F}(V)$.

For the free-electron model in the case of a cubic lattice [4], the Fermi energy is determined as follows:

$$E_F = \frac{\hbar^2}{2m}\left(\frac{3\pi^2 N}{V}\right)^{\frac{2}{3}}, \tag{2}$$

where m is the mass of an electron; N, number of free electrons; V, volume of an elementary cell.

For the strongly bound-electron model, in the case of a triclinic lattice, including different atoms, no analytic expression was found for the Fermi energy. Consequently it does not seem possible as yet to use the expression found in [1] to estimate the deformations of the crystal lattice in kaolinite during the adsorption of various substances onto its surface. We used an approach proposed by A. M. Stoneham [5] and based on a consideration of the various contributions made to the distortion of the lattice by donor or acceptor defects. In real crystals the elastic deformation of the volume of an elementary cell far from a point defect, according to [5], can be determined as follows:

$$\frac{\Delta V}{V} = \left(\frac{B}{r^3} + \frac{B'}{r^5}\right)\left(\frac{x^4 + y^4 + z^4}{r^4} - 0.6\right), \tag{3}$$

where B is the term arising as a result of the anisotropic elasticity. For low anisotropy it is proportional to the expression

$$B = \frac{375}{8\pi} \cdot \frac{(c_{11} + 2c_{12})(c_{11} - c_{12} - 2c_{44})}{(3c_{11} + 2c_{12} + 4c_{44})^2}\, \Delta V_1. \tag{4}$$

The second term B' appears as a result of the fact that the forces in the direction of the defect are applied not to a point but to a volume of radius R, which is approximately equal to the distance between its nearest neighbors:

$$B' = R^2 \frac{105}{32\pi} \sqrt{\frac{3}{2}}\left(\frac{c_{11} + 2c_{12}}{c_{11}}\right)\Delta V_1. \tag{5}$$

In the above formulas the quantities c_{11}, c_{12}, c_{44} are the elastic moduli of the crystal; x, y, z, coordinates of the defect; r, distance from the center of gravity of the crystal to the defect; ΔV_1, defined in [5] as the difference between the volume per atom of the main crystal and the volume of an atom of the element of Group IV with tetrahedral symmetry that is closest in atomic number to the donor.

According to the electronic theory of catalysis [3], the chemisorbed particle is regarded as an "impurity" which has penetrated into the surface of the crystal or as some structural defect which disturbs the strictly periodic structure of the surface. In this treatment, which was first mentioned in [6], the chemisorbed particle and the lattice of the adsorbent are represented as a single quantum-mechanical system, and it is automatically ensured that the chemisorbed particles participate in the electron economy of the lattice. In [7, 8] it was shown that a chemisorbed particle, regarded as a structural defect of the surface, becomes a localization center for a free electron of the lattice and serves as a trap for it, acting the role of an acceptor, or (this depends on the nature of the particle) it may serve as a localization center for a free hole, acting the role of a donor.

Thus, the approach used in [5] can be used in our case to estimate the deformations of the crystal lattice. At the same time, it is clear that we cannot calculate $\Delta V/V$, not knowing the moduli of elasticity of the crystal, but we can approach the solution of the problem in a somewhat different manner.

It can easily be shown that the ratio of the change in volume of a crystal lattice for an arbitrary intermediate stage of sorption to the change in the volume during the initial period of sorption can be represented in the following manner:

$$\frac{\Delta V}{\Delta V_0} = \frac{\sum_{i=1}^{k} \frac{1}{r^3} \left(\frac{x^4 + y^4 + z^4}{r^4} - 0.6 \right) \left(\frac{R_J}{R_0} \right)^3}{\sum_{i=1}^{n} \frac{1}{r^3} \left(\frac{x^4 + y^4 + z^4}{r^4} - 0.6 \right) \left(\frac{R_J}{R_0} \right)^3},$$ (6)

where i is the number of defects (atoms of adsorbent) entering into a bond with active centers of the surface at different stages of sorption; R_0, radius of one of the sorbed atoms; and R_J, radii of the other sorbed atoms.

For subsequent calculations on the basis of the measurements of the specific surface and an estimate of the dimensions of the microaggregates from electron photomicrographs, we selected the dimensions of the crystal. According to [9], we take the following parameters of an elementary spatial cell of the crystal: $a = 5.14$ Å; $b = 8.93$ Å; $c = 7.37$ Å; $\alpha = 91.8°$; $\beta = 104.7°$; $\gamma = 90°$, and the calculated crystal under consideration was composed of $2.9 \cdot 10^6$ elementary spatial cells with over-all dimensions of 353 Å; 712 Å; 488 Å (the coordinates of the center of gravity were $x_0 = 176.72$ Å; $y_0 = 356.44$ Å; $z_0 = 243.87$ Å). For the origin we took the point at the intersection of the edges of a spatial cell passing through oxygen atoms O_1 and O_8. A diagram of the arrangement of the atoms in a cell is shown in Fig. 2.

Let us estimate the change in the quantity $\Delta V/\Delta V_0$ for some elementary volume with the coordinates of the center of gravity of the crystal in the process of simultaneous sorption of water and toluene, water and nitrobenzene, and water alone.

It is easily shown that the coefficients $K = [(x^4 + y^4 + z^4)/r^4 - 0.6]/r^3$ are practically the same for the atoms of a single cell. Consequently, in order to observe the variation of $\Delta V/\Delta V_0$, it is sufficient to consider the influence of defects situated only in the six cells shown in Table 1.

A fragment of the calculation scheme is shown in Table 1 and reduces to successive summation of the contributions (the quantities $K(R_J/R_0)^3$) of the adsorbed atoms of the toluene molecules, the water molecules, and the nitrobenzene molecules. We have tentatively used a + before the coefficients K for the acceptor atoms of the crystal lattice and a — for the donor atoms. Here we must point out that in formula (6) the quantity taken for R_0 is the metallic radius of the carbon atom [10], and for the R_J we use the radii of the other atoms (oxygen, hydrogen, nitrogen).

Let us observe the change in the sums of the coefficients $K(R_J/R_0)^3$ in Table 1. The first columns of the table refer to the adsorption of toluene on the dehydrated surface. In this stage, on the 100 planes, we take as the centers of adsorption the hydroxyl groups $H_{12}(O_{12})$ and $H_{13}(O_{13})$, arranging on these the diametrically opposite carbon atoms of the benzene ring, on the 010 planes we take as the centers of adsorption the groups $H_{16}(O_{16})$, and on the 001 planes we take $H_6(O_6)$, $H_5(O_5)$, O_5, and O_6. In this case the benzene ring is parallel to the surface, and the toluene molecules appear in the interaction with the hydroxyl groups or oxygen atoms by the mechanism of π-bonds [11, 12]. As can be seen from
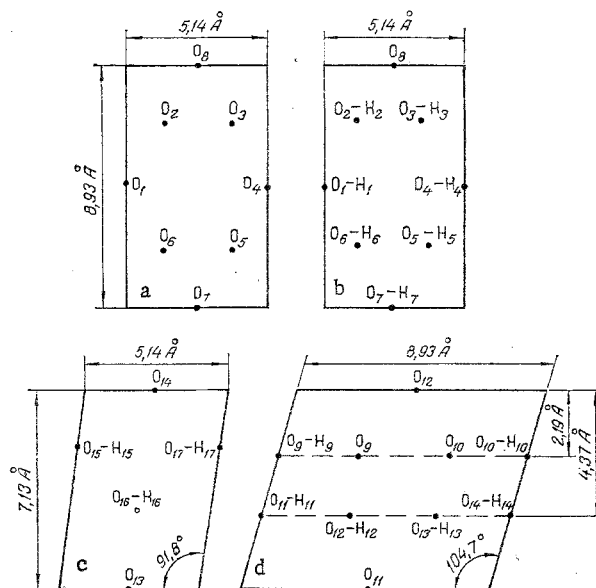
Fig. 2. Arrangement of atoms in the planes of
an elementary cell of kaolinite: a and b) 001
plane; c) 010 plane; d) 100 plane.

Fig. 1, under the influence of the toluene molecules there is an expansion of the spatial
cell in comparison with the purely dehydrated crystal or in the adsorption of nitrobenzene
molecules. This expansion is taken to be the initial state, and it is with respect to this
that we estimate the change in volume both in the adsorption of water and toluene and in the
adsorption of water or of water and nitrobenzene.

In the first stage of hydration (P = 0.1-0.2%) the water molecules are rigidly attached
to the surface at the centers of the 100 plane. In this case the toluene molecules are
bound to the surface through free hydroxyl groups $H_{11}(O_{11})$, $H_{13}(O_{13})$, with vertical orienta-
tion of the plane of the benzene ring with respect to the surface of the crystal. The π-
bond changes to a σ-bond [11]. The number of toluene molecules adsorbed on the six plane
cells increases from six to eight in comparison with the initial state.

In the second stage (P = 0.3-0.5%) the water molecules are arranged, in addition, on
the centers of $H_{12}(O_{11})$ and O_{11} in the 100 planes, and on $H_{15}(O_{15}) - O_{14}$, $H_{16}(O_{16}) - O_{13}$ in
the 010 planes. The toluene molecules adsorbed on the centers of $H_{13}(O_{13})$ and $H_{17}(O_{17})$ near
the water molecules enter into a π-complex interaction with them, making an additional con-
tribution to the deformation of the crystal lattice.

In the third stage (P = 0.7-1.1%) the water molecules form dimers on the 100 and 010
planes, i.e., they are now adsorbed on the bound molecules of H_2O and are also arranged on
the 001 planes. As a result of the appearance of single bonds, an unstable water structure
arises. There is an increase in the effect of π-complex interaction, which results in maxi-
mum deformation of the crystal lattice.

In the fourth stage of hydration (P = 1.2-2.4%) we observe successively the formation
of dimers and the adsorption of water molecules on vacant OH groups, the displacement of
toluene molecules from the surface of the crystal, and, as a result, a gradual reduction in
the contribution to the deformation of the crystal lattice.

As can be seen from Fig. 1, the maximum deformations of the lattice under the influence
of water and toluene (curve 1) appear at a 0.9-1% water content, and in the subsequent part
of the process the sorptions gradually decrease. At a moisture content of 2.4-2.5%, curve 1
degenerates into curve 2, which characterizes the deformation as a result of water adsorption
alone. This very important experimental result confirmed (in another way) the existence of
a second effect: the change in the density of the dispersion medium in the system consisting
of a solid, adsorbed water, and liquid. The adsorption of the toluene molecules is the re-
sult of the specific interaction of the π-bonds of the benzene ring with the hydroxyl groups
of the crystal lattice. In such an interaction the hydroxyl groups are electron acceptors.
And, according to [12], the redistribution of the charge in the benzene molecule and the

TABLE 1. Calculation of the Sum of the Coefficients $(R_J/R_0)^3 K$ and the Coefficients of Relative Change in Volume of an Elementary Cell of Kaolinite in the Process of Adsorption of Water and Toluene

Plane	No. of planes	Adsorbed molecules	No. of molecules	Atoms of lattice	$\left(\dfrac{R_J}{R_0}\right)^3 K\cdot10^4$
100	1	$C_6H_5CH_3$ *	1	$H_{12}(O_{12})$ $H_{13}(O_{13})$	+5,2322 +5,1265
	1	$C_6H_5CH_3$ *	1	$H'_{12}(O'_{12})$ $H'_{13}(O_{13})$	+7,4483 +7,2378
010	1	$C_6H_5CH_3$ *	1	$H'_{16}(O'_{16})$	+0,9263
	1	$C_6H_5CH_3$ *	1	$H_{16}(O_{16})$	−0,0020
001	1	$C_6H_5CH_3$ *	1	$H_6(O_6)$ $H_5(O_5)$	+0,3965 +0,3984
	1	$C_6H_5CH_3$ *	1	O_6 O_5	−0,1125 −0,1133
				ΣK_1	26,5382
				$P,\%$	0,00
				n_t	6
				$\Delta V : \Delta V_{0P}$	1,00
				$\Delta V : \Delta_0 eq$	1,00

Plane	No. of planes	Adsorbed molecules	No. of molecules	Atoms of lattice	$\left(\dfrac{R_J}{R_0}\right)^3 K\cdot10^4$
100	1	H_2O	1	$H_9(O_9)$ O_9	+4,803 −3,193
		H_2O	1	$H_{10}(O_{10})$ O_{10}	+4,5914 −3,1409
		$C_6H_5CH_3$	2	$H_{11}(O_{11})$ $H_{13}(O_{13})$	+4,6650 +5,1265
010	1	$C_6H_5CH_3$ *	1	$H'_{12}(O'_{12})$ $H'_{13}(O_{13})$	+7,4483 +7,2378
	1	$C_6H_5CH_3$ *	1	$H'_{16}(O'_{16})$	+0,9263
001	1	$C_6H_5CH_3$ *	1	$H_{16}(O_{16})$	−0,0020
	1	$C_6H_5CH_3$ *	1	$H_6(O_6)$ $H_5(O_5)$	+0,3965 +0,3984
	1	$C_6H_5CH_3$ *	1	O_6 O_5	−0,1125 −0,1133
				ΣK_1	+29,0588
				$P,\%$	0,1
				n_t	7
				$\Delta V : \Delta V_{0P}$	1,0948
				$\Delta V : \Delta V_0 eq$	1,0862

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Table (rotated):

Molecule	Count	Position	Label	Values
H₂O	1		$H_9(O_9)$ / O_9	+4,803 / −3,193
H₂O	1		$H_{10}(O_{10})$ / O_{10}	+4,5919 / −3,1409
H₂O	1	100	$H_{12}(O_{12})$ / O_{11}	+4,7942 / −3,2020
H₂O	2		On adsorbed H₂O [12, 9]	
C₆H₅CH₃	1		$H_{11}(O_{11})$ / $H[H_2O]$ [9]	+4,6650 / +4,3000
C₆H₅CH₃	1		$H_{13}(O_{13})$ / $2H(H_2O)$ [12]	+5,1265 / +9,5356
H₂O	1		$H_9'(O_9')$ / O_9'	+6,8996 / −4,4677
H₂O	1		$H_{10}'(O_{10}')$ / O_{10}'	+6,8318 / −4,2754
H₂O	1		$H_{12}'(O_{12}')$ / O_{11}'	+6,8318 / −4,5220
H₂O	2		On adsorbed H₂O [12, 9]	
C₆H₅CH₃	1		$H_{13}'(O_{13}')$ / $2H(H_2O)$ [9]	+7,2378 / +12,5244
C₆H₅CH₃	1		$H_{11}'(O_{11}')$ / $H(H_2O)$ [9]	+6,6890 / +7,4000

Molecule	Count	Position	Label	Values
H₂O	1		$H_{16}'(O_{16}')$ / O_{13}	+0,8615 / −0,5773
H₂O	1		$H_{15}'(O_{15}')$ / O_{14}	+0,8615 / −0,5773
H₂O	1	010	On adsorbed H₂O [15]	
C₆H₅CH₃	1		$H_{17}'(O_{17}')$ / $2H(H_2O)$ [15]	+0,9053 / +1,7728
H₂O	1		$H_{16}(O_{16})$ / O_{13}	−0,0018 / +0,0013
H₂O	1		$H_{15}(O_{15})$ / O_{15}	−0,0018 / +0,0013
C₆H₅CH₃	1		$H_{17}(O_{17})$ / $H(H_2O)$ [15]	−0,0021 / −0,0020
H₂O	1		$H_2(O_2)$	+0,3550
H₂O	1	001	$H_6(O_6)$	+0,3600
C₆H₅CH₃	1		$H_{13}(O_{13})$ / $2H(H_2O)$ [2]	+0,3871 / +0,8000
H₂O	1		O_3 / O_5	−0,0664 / −0,0670

ΣK_1	75,2887
P, %	0,9
n_t	7
$\Delta V : \Delta V_{0P}$	2,8449
$\Delta V : \Delta V_{0eq}$	2,9310

Remark. An asterisk indicates a horizontal position of the toluene molecules; the other cases correspond to a vertical position of the molecules.
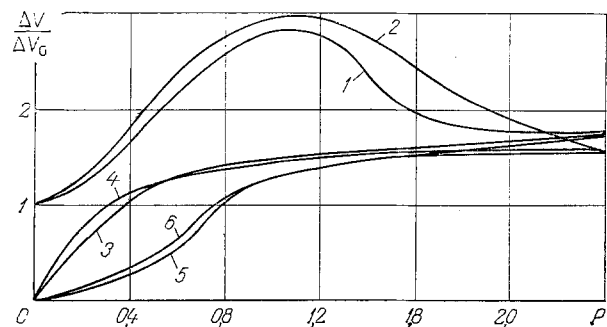
Fig. 3. Relative change in the volume of an elementary cell of the natural form of Glukhovetsk kaolinite, obtained from experimental curves and from calculations when the liquid adsorbed is: 1, 2)ʹ water and toluene; 3, 4) water; 5, 6) water and nitrobenzene.

molecules of other alkylbenzenes (including toluene), as well as the associated variation in the dipole moments, especially in the case of underfilled hydroxylized surfaces, differs sharply from the corresponding changes in ordinary intermolecular interactions in the liquid state and from the corresponding distribution in the absence of any interactions in the gaseous state. Consequently the toluene molecules (in our case) compete (and this is reflected in the calculation schemes) with the water molecules for the active centers of the surface.

In the calculation schemes the adsorption of water molecules by single bonds only on the OH groups of the surface (weak interaction) is provided for in the case of relatively high filling values (P = 1-2.5%), when the toluene molecules are displaced practically completely from the surface layer. It should be noted that in estimating the relative changes in the volumes of an elementary cell of kaolinite when only water is adsorbed, the order in which the active centers are filled is fundamentally analogous to the order followed when there is adsorption of water and toluene, but in this case even in the early stages of sorption there is attachment of the molecules of water only to the OH groups of the crystal-lattice surface.

Let us consider the deformation of the crystal lattice under the influence of water and nitrobenzene. The π-electron distribution in the ground state of $C_6H_5NO_2$ taken from [13] was described by us in [14].

Consequently the adsorption of the nitrobenzene is due both to the transfer of the charge to the crystal-lattice surface — and in this case the electron donors in the molecule will be the oxygen atoms or the carbon atom situated adjacent to the NO_2 group, while the acceptors on the crystal lattice will be the OH groups — and to the pulling away of electrons from the solid phase, in which case the electron acceptors in the molecule will be the nitrogen atom and the other carbon atoms, while the donors on the crystal lattice will be the oxygen atoms.

In the case of complete dehydration, what we take as the centers of adsorption of nitrobenzene on the 100 planes are the hydroxyl group $K_{12}(O_{12})$ and the oxygen O_{11}, placing on these the diametrically opposite carbon atoms of the benzene ring; on the 010 planes, we take $H_{16}(O_{16})$ and O_{13}, and on the 001 planes, $H_2(O_2)$, $H_3(O_3)$, $H_6(O_6)$, $H_5(O_5)$, O_6 and O_5.

It is easy to show that the sum of the coefficients $K(R_J/R_0)^3$ in this case, and consequently the initial deformation ΔV_1 of a dehydrated spatial cell as well, will be close to zero. This result is confirmed by experimental measurements (see Fig. 1).

In the first stage of hydration (P = 0.2%) the water molecules are rigidly attached to the surface on the centers of the 100 plane. Here too, it must be pointed out that the order in which the active centers are occupied by the water molecules at this stage and subsequent stages is taken to be analogous, as in the case of adsorption of water and toluene. The nitrobenzene molecules are bound to the surface through free hydroxyl groups $H_{11}(O_{11})$, $H_{13}(O_{13})$, O_{11}, O_{12}, orienting themselves vertically along the plane of the benzene ring with respect to the crystal surface.

The number of nitrobenzene molecules adsorbed on the six plane cells is increased from six to ten.

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In the second stage (P = 0.4%) and the third stage (P = 0.6%) of hydration the water molecules are arranged, in addition, on the centers $H_{12}(O_{12})$ and O_{11} in the 100 planes; on $H_{15}(O_{15}) - O_{14}$, $H_{16}(O_{16}) - O_{13}$ in the 010 planes; and on O_2, O_6, $H_7(O_7)$ in the 001 planes. The nitrobenzene molecules gradually give up their positions on the 100 and 010 planes and take up positions on the 001 planes, orienting themselves vertically, and their contribution to the deformation has a different sign depending on the structure of the active centers.

The maximum number of nitrobenzene molecules on one spatial elementary cell is equal to 12, which corresponds to a moisture content of 0.4%.

The subsequent stages of hydration (P = 0.8-2.5%) result in the gradual occupation of the active centers by water molecules and the displacement of the nitrobenzene from the surface layer of the crystal lattice.

Figure 3 shows the values of $\Delta V/\Delta V_0$ taken from the experimental curves 1, 2, 3 (see Fig. 1) and those obtained from calculations. The close agreement between the experimental and theoretical values gives some assurance that the idea and the scheme of the calculation were correctly chosen. Here too, it must be noted that in the calculation schemes we considered an ideal crystal surface, i.e., we took account only of the active centers represented by the hydroxyl groups and the oxygen atoms and did not take account of the cleavage segment, when uncompensated Si and Al atoms appear on the outside.

In determining the relative changes in the volume of an elementary cell of kaolinite, the effect of different atoms of adsorbed molecules was estimated on the basis of a calculation of their coordinates, taking account of the direction of electron transfer and the atomic radii.

At the same time, the fraction of the charge transferred to the crystal or to the adsorbed molecule is determined in large measure by the interatomic interaction of adjacent molecules. More detailed investigations of the topography of the crystal-lattice surface, carried out by other methods, and an estimate of the charge transfer by the method of electron paramagnetic resonance apparently make it possible to perform the calculations more reliably and more accurately.

However, the results obtained with regard to the deformations of the crystal lattice under the influence of water, toluene, and nitrobenzene have provided us with some new ideas concerning the structure of adsorbed water and enabled us to correct the value of its density.

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