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DIAGNOSTICS OF THE STRUCTURE OF CRYSTALLINE MATERIALS OF ROCK FORMATIONS BY THE METHOD OF THERMALLY STIMULATED DEPOLARIZATION

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Abstract. Relevance. Crystalline materials in rocks undergo structural changes under high pressure and in humid or aggressive environments, and also during ship or land transport at both high and low temperatures. These conditions generate defects and alter their concentration, demanding reliable diagnostics and monitoring. Objective. To study defect structure and hydration state in rock-formation crystalline materials—gypsum dihydrate $CaSO_4 \cdot 2H_2O$, copper sulfate pentahydrate $CuSO_4 \cdot 5H_2O$, and magnesium hydrosilicate $Mg_3Si_4O_{1\,02}$ - using thermo-stimulated depolarization currents (TSDC), and to develop diagnostics applicable to insulation materials and mining operations. Methods. We measured TSDC spectra of natural and calcined crystals at different temperatures, including samples doped with alkali (NH_4OH) and acid (HCl), using a patented apparatus. From these spectra we determined activation energies, defect types, and defect concentrations in the 77–473 K range at current densities from 10^{-6} to 10^{-15} A/m². We propose a TSDC-based method to determine the number of water molecules in a crystal's formula unit and computed diffractograms for calcium and copper sulfate hydrates with varying water content. Results and Conclusions. Changes in the position and magnitude of TSDC maxima relative to reference spectra, especially in alkaline or acidic media, provide a basis for quality diagnostics of crystalline materials and enable ecological monitoring during extraction and transportation. The approach yields key defect parameters and hydration counts, with results demonstrated for $CaSO_4 \cdot 2H_2O$ and $CuSO_4 \cdot 5H_2O$.

Keywords: crystalline materials, rock formations, thermostimulated depolarization currents, diagnostics, diffractograms, environmental monitoring

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ТЕРМОСТИМУЛЯЦИЯЛАНҒАН ДЕПОЛЯРИЗАЦИЯ ӘДІСІМЕН ТАУ ЖЫНЫСТАРЫНЫҢ КРИСТАЛДЫҚ МАТЕРИАЛДАРЫНЫҢ ҚҰРЫЛЫМЫН ДИАГНОСТИКАЛАУ

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Аннотация. Өзектілігі. Минералдардың кристалдық құрылымындағы өзгерістер тау жыныстарында жоғары қысымның, температураның және агрессивті ортаның әсерінен, сондай-ақ материалдарды теңіз және құрлық арқылы тасымалдау кезінде болады. Бұл процестер кристалды материалдардың физика-химиялық және пайдалану қасиеттеріне айтарлықтай әсер ететін құрылым ақауларының пайда болуына және концентрациясының өзгеруіне әкеледі. Мақсат. Жұмыстың мақсаты кальций сульфаты кристаллогидраты ($CaSO_4 \cdot 2H_2O$), мыс ($CuSO_4 \cdot 5H_2O$) және магний гидросиликаты ($Mg_3[Si_4O_{10}][OH]_2$) сияқты кристалдық материалдардың құрылымдық ақаулары мен гидратациясын эксперименттік және теориялық зерттеу болып табылады. Олардың сапасын диагностикалау әдістерін әзірлеу және олардың кристалдық құрылымына сыртқы факторлардың әсерін бағалау. Әдістері. Зерттеулер патенттелген қондырғыда термостимуляцияланған деполяризация токтары (TCD) әдісін қолдана отырып жүргізілді. Табиғи және әртүрлі температурада кальциленген кристалдар, сондай-ақ сілтімен (NH_4OH) және қышқылмен (HCl) легирленген үлгілер талданды. Активтендіру энергиясы, 10^{-6}–10^{-15} А/м^2 ток тығыздығы кезінде 77-473К температура диапазонындағы құрылым ақауларының түрлері мен концентрациясы анықталды. Сонымен қатар, формула бірлігінде су молекулаларының әртүрлі құрамы бар кристалды гидраттарға арналған дифрактограммаларды есептеу жүргізілді. Нәтижелер мен қорытындылар. Термостимуляцияланған деполяризация деректері бойынша зерттелетін кристалдардың формула бірлігіндегі су молекулаларының санын анықтау әдісі ұсынылған. TSTD максимумдарының орналасуы мен амплитудасының үлгілердің құрамы мен күйіне тән тәуелділігі анықталды, бұл кристалды материалдардың сапасын диагностикалау критерийлерін жасауға мүмкіндік берді. Сілтілік немесе қышқылдық ортадағы TCD максимумдары қарқындылығының өзгеруі тау жыныстарының кристалды материалдарын өндіру және тасымалдау ортасын экологиялық бақылау үшін, сондай-ақ олардың электр оқшаулау және тау-кен технологияларында жарамдылығын бағалау үшін пайдаланылуы мүмкін екендігі көрсетілген.

Түйін сөздер: кристалдық материалдар, тау жыныстары, термостимуляцияланған деполяризация токтары, диагностика, дифрактограммалар, экологиялық мониторинг

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ДИАГНОСТИКА СТРУКТУРЫ КРИСТАЛЛИЧЕСКИХ МАТЕРИАЛОВ ГОРНЫХ ПОРОД МЕТОДОМ ТЕРМОСТИМУЛИРОВАННОЙ ДЕПОЛЯРИЗАЦИИ

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Аннотация. Актуальность. Изменения кристаллической структуры минералов в горных породах происходят под воздействием высокого давления, температурных градиентов, химически активных сред, а также в процессе транспортировки материалов морским и наземным транспортом. Эти факторы вызывают образование и перераспределение структурных дефектов, существенно влияющих на физико-химические и эксплуатационные свойства кристаллических материалов. Цель. Цель исследования — экспериментальное и теоретическое изучение дефектов структуры и процессов гидратации кристаллических материалов на примере кристаллогидратов сульфатов кальция ($CaSO_4 \cdot 2H_2O$), меди ($CuSO_4 \cdot 5H_2O$) и гидросиликата магния ($Mg_3[Si_4O_{10}]$

$[OH]_2$), а также разработка диагностических методов оценки их качества и устойчивости к воздействию внешних факторов. Методы. Исследования выполнены методом термостимулированных токов деполяризации (ТСТД) на запатентованной экспериментальной установке. Анализу подвергались природные и прокалённые образцы, а также кристаллы, легированные щелочью (NH_4OH) и кислотой (HCl). Определены энергия активации, типы и концентрации дефектов в диапазоне температур 77–473 К при плотности тока 10^{-6}–10^{-15} А/м². Дополнительно рассчитаны дифрактограммы кристаллогидратов с различным содержанием воды во формульной единице. Результаты и выводы. Предложен метод количественного определения числа молекул воды в формульной единице кристалла по данным ТСТД. Установлены характерные зависимости положения и амплитуды максимумов ТСТД от состава, структуры и состояния образцов, что позволило сформулировать критерии диагностики качества кристаллических материалов. Показано, что смещение и изменение интенсивности максимумов ТСТД в щелочной и кислотной среде могут служить индикаторами экологического состояния среды добычи и транспортировки, а также использоваться при оценке пригодности материалов для электроизоляционных и горнодобывающих технологий.

Ключевые слова: кристаллические материалы, горные породы, термостимулированные токи деполяризации, диагностика, дифрактограммы, экологический мониторинг

Introduction. Physical methods of rock research play an important role in the development of geophysical methods (Bosikov, Klyuev, Silaev, Stas, 2023). The proposed diagnostics are based on a patent and belong to non-destructive testing methods, and can also be used for physical and chemical analysis of rocks and in the control of the chemical composition of crystalline and electrical insulating materials (Malozyomov et al., 2025).

The purpose of the study is to improve the accuracy, reliability and environmental safety in determining the structure in a wider range of crystalline materials as a result of studying and comparing the spectra of thermally stimulated depolarization currents (TCTD) of natural, as well as doped and calcined samples, which makes it possible to determine the concentration of structural defects and the presence of a certain type of water molecules in the formula unit of rock crystals (Nussipali et al., 2024, Pryalukhin et al., 2024). For this purpose, the maxima of the TSTD spectrum of samples calcined at temperatures corresponding to the yield of a certain type of water molecules are investigated, which significantly reduces these maxima, while the ratio of the maximum densities of the maxima No 3 of the TSTD of the reference and the studied crystalline materials is directly proportional to the ratio of the number of crystallization water molecules in the formula unit, the absence of these maxima indicates their complete dehydration (Bosikov, Klyuev, Khetagurov, Silaev, 2023; Martyushev et al., 2025, Pryalukhin et al., 2024). Changes in the position and magnitude of other maxima indicate a change in the environmental situation, which makes it possible to carry out environmental monitoring (Morgoeva et.al., 2024; Shabanov et al., 2023).

Materials and equipment. In the study of TSTD, a patented multifunctional device was used to study the physical and technical characteristics of materials at temperatures (77... 473) K and frequencies of the electric field (1... 10^8) Hz. The increase in accuracy is provided by the use of precision equipment with an accuracy of current measurement up to 10^{-15} A and the use of a double-shielded cable. Reliability is achieved by the use of the TCTD method, as well as the simplicity and reliability of the method. The device (fig. 1) allows you to measure tgδ from units to 10^{-4}, current intensity up to 10^{-15} A, electric capacitance up to 1 picofarad, electric field strength up to breakdown values, resistances up to 10^{-18} Ohm, take temperature spectra of TSTD in the temperature range (77... 473) K and higher with the help of liquid nitrogen. Inside the plant, a pressure of about 10^{-4} mm Hg was provided by means of backing and diffusion pumps (Martyushev et al., 2025). The environmental safety of the proposed method is much higher than with the chemical and X-ray diffraction methods.

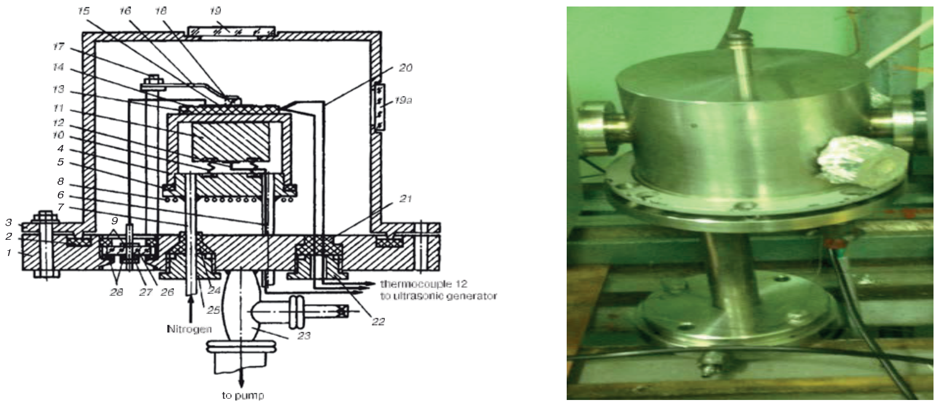
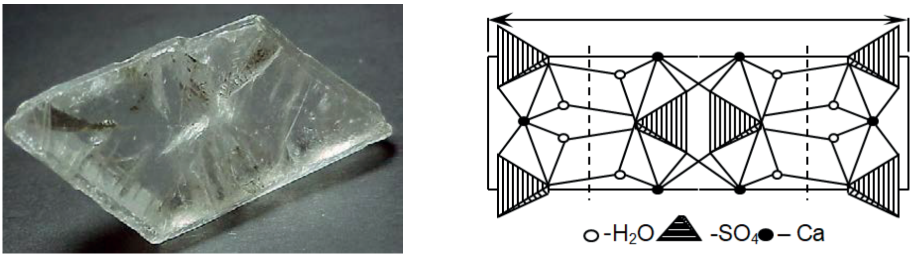


Fig. 1. Design and appearance of the pilot plant

Natural crystal hydrates of calcium sulfate $CaSO_4$ were selected for the research$\cdot 2H_2O$ and copper pentahydrate $CuSO_4 \cdot 5H_2O$ (triclinic lattice), as well as layered crystals of magnesium hydrosilicate $Mg_3[Si_4O_{10}]$ $[OH]_2$, (monoclinic syngony, point group 2/m is prismatic, cleavage is very perfect in {001}, imperfect in {110} and {010}) (Figs. 2, 3, 4). These crystals are united by the fact that they have hydrogen bonds to a greater or lesser extent. Gypsum (calcium sulfate) was supplied in the form of druses or blocks of crystals from the Zhairem deposit in the Dzhezkazgan region. Crystals with a thickness of (20... 25) mm were easily split into thin plates along cleavage planes {010} thickness (0.3... 1) mm, after which they were polished with sandpaper, grinding powder and grinding glasses to the desired dimensions and polished with Goya paste.

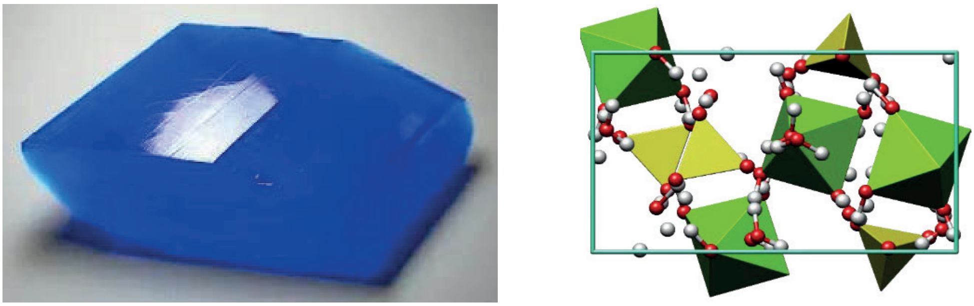
At a temperature of 120°C, calcium sulfate loses 3/4 of its water, turning into alabaster. And at 160°C, semi-aqueous calcium sulfate (β-hemihydrate) is formed. At 170°C, water is lost β hemihydrate, and at 325°C, anhydrite is formed. Differential thermal analysis of crystals was carried out on a Q – 1000 Derivatograph.



a b

Fig. 2. Samples of calcium sulfate crystal hydrate $CaSO_4 \cdot 2H_2O$: a – appearance, b – structure

Chalcanthite or copper pentahydrate $CuSO_4 \cdot 5H_2O$, like calcium sulfate, has a layered crystallographic structure (fig.3), which includes CuO – 31.8%, SO_3 – 32.1%, H_2O – 36.1%. When heated, it gradually loses water, turns first into three-aqueous sulfate, and then into monoaqueous sulfate, while becoming white and opaque. A single crystal of copper sulfate pentahydrate occurs in nature in the form of the minerals chalcanthite ($CuSO_4 \cdot 5H_2O$), chalcokyanite ($CuSO_4$), bonattite ($CuSO_4 \cdot 3H_2O$), butite ($CuSO_4 \cdot 7H_2O$). Two SO_4^{2-} anions along the axes and four water molecules (in the plane) are coordinated around the copper ion, and the fifth water molecule acts as bridges that unite the water molecules from the plane and the sulfate group with the help of hydrogen bonds.



a b

Fig. 3. Samples of copper sulfate crystal hydrate $CuSO_4 \cdot 5H_2O$: a – appearance, b – structure

When heated, the pentahydrate sequentially splits off two water molecules, turning into trihydrate $CuSO_4 \cdot 3H_2O$, then into monohydrate (at 110 °C) $CuSO_4 \cdot H_2O$, and above 258°C, the anhydrous salt $CuSO_4$ is formed, which becomes white and opaque. After their processing and drying, the spectra of the CTTD were taken.

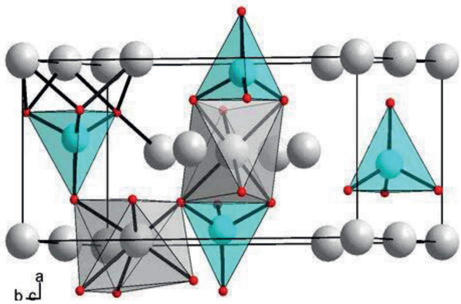
A huge number of minerals belong to the class of silicates. They account for about one-third of the number of minerals known in nature. Silicates make up 75% of the earth's crust, from which the exceptional role they play in nature can be seen. A characteristic representative of the class of silicates is Onot talc, a natural magnesium hydrosilicate $Mg_3(Si_4O_{10})(OH)_2$ or $3MgO·4SiO_2·H_2O$, which exists in the form of leafy, less often lamellar crystals. The basis of the crystal structure is tetrahedral orthogroups $[Si_4O_{10}]^{4-}$ with ribs 0.26 nm long, which, when combined, form two-dimensional layers of hexagonal rings with the main structural unit $[SiO_4]^{4-}$. The monoclinic talc lattice is built on hexagonal grids Si_4O_{10}, which armor a layer of brucite $Mg(OH)_2$ on both sides and form three-layer packages (fig. 4, table 1). The structure of brucite provides the densest packing of OH^- ions

Onot talc was supplied in the form of dense white packages, blocks. They were easily sawn along the cleavage plane, sanded on sandpaper of various grains and finished on grinding glasses. At the same time, the parallelism of the sides of $\pm 2 \times 10^{-6}$ m was achieved with a thickness of 1... 3 mm, which ensures good contact with the overhead electrodes. In addition, electrodes (silver, aluminum) were applied to the samples by thermal vacuum deposition at the VUP-5 facility. Conductive lubricant was also used. The samples were calcined in a muffle furnace at various temperatures required for the experiment for eight hours at a cooling rate of no more than 20 °C/h. The most stable and reliable data with good peak separation for all crystals were obtained with steel overhead electrodes and adhesive electrodes based on AK-113 glue and nickel powder.

In addition to hydroxyl water, talc crystals contain molecular water, its content ranges from 2.9 to 6%. Talc dehydration occurs in three stages. At 100°C, adsorbed water begins to be released, at 575°C – structural water, at (950... 960)°C – constitutional, When firing over 1400 °C, clinoenstatitis is formed. In the interval (700...1000)°C, there is a weakening of bonds. In minerals, water molecules are located between layered packages with weak residual bonds, which, for crystallochemical reasons, should be located in these layers in a strictly oriented manner.

Table 1. Chemical composition of magnesium hydrosilicate crystals (%)

SiO_2	Al_2O_3	Fe_2O_3	FeO	CaO	TiO_2	MgO	K_2O	N_2O	H_2O
62,2	1,25	0,65	0,32	0,1	0,44	32,2	0,1	0,1	3



a b

Fig. 4. Magnesium hydrosilicate samples $Mg_3(Si_4O_{10})(OH)_2$: a – appearance, b – structure

Results and discussion. In the study of the CTT spectra of the crystals mentioned above, it was found that the maximum of 3 decreases only at calcination temperatures corresponding to the change in the crystal structure (Zemlyanoy et. al., 2024). At the same time, the water content in the formula units of crystals changed. The height of this maximum TSTD turned out to be proportional to the number of water molecules in the formula unit of crystal hydrates. Therefore, it was concluded that the maximum of 3 in crystal hydrates, as well as in silicates, It is caused by the relaxation of crystallization (or structural) water molecules. The absence of this maximum in the TSTS spectrum indicates the absence of crystallization water molecules both in sulfates (curve 5, fig. 7), copper sulfate (curve 4, fig. 9), and in the TSTD spectrum of magnesium hydrosilicate crystals $Mg_3[Si_4O_{10}][OH]_2$, where, after calcination at a temperature above 1323 K, a maximum of 3 is practically absent (curve 4, fig. 10), which corresponds to its transformation into steatite, from which the insulating material steatite ceramics is made. For all the crystals studied, the activation energy was determined using the Garlick-Gibson initial ascent method by partial heat treatment (tables 2, 3, 4).

The method of the initial rise in determining the activation energy is based on the fact that at low temperatures in the initial section, the DCT current curves have the form . From this it can be seen that the initial section of the DCT current in Arrhenius coordinates ($\ln I \sim 1/kT$) is a straight line, which makes it possible to graphically determine the activation energy of electrically active defects.

Partial heat treatment method. In the crystalline materials studied by us, the number of defects reaches 5–6 types. This means that a number of maxima are superimposed on the TSTD spectrum. To isolate each maximum, it is necessary to suppress the low-temperature maximum preceding the studied one It is then cooled to a temperature at which the current drops to zero. After that, heating is carried out at the same rate and the low-temperature maximum disappears, forming the initial rise of the next maximum, which makes it possible to calculate the activation energy of the defect (fig. 5, table 2–4). The sample is then heated to a temperature 10 K higher than the temperature of the next maximum, cooled again and the next maximum is extracted. The process of partial thermal cleaning was performed by us several dozen times for various materials, and the result was invariably repeated for each maximum with an accuracy of (3...5) %.

For copper pentahydrate, as well as for calcium sulfate crystal hydrate and magnesium hydrosilicate, the activation energy was determined after partial thermal treatment and the separation of all maximums. By comparing the activation energies and temperature of the ice maxima and the crystals under study, it is possible to conclude about the types of relaxers responsible for the appearance of the TSTD maxima, which is shown in Table 2–4.

To reveal the nature of relaxation of other maxima, a special technique was developed in which certain types of defects were activated, while others did not change their parameters. First, samples calcined at different temperatures were studied. Then natural samples were placed in HCl and NH_4OH solutions, where they were kept for a long time (up to 100 days) or grown in doped solutions. after drying and cleaning the surface of the samples, the CTT spectra were taken (Fig. 6–9).

Both talc and pyrophyllite $Al_2[Si_4O_{10}][OH]_2$ are characterized by diffuse scattering of X-rays, which indicates the existence of defects in the packing of the layers. Connecting double layers are completely absent, which causes perfect cleavage, a very high ability to split and significant softness of these minerals. In addition, partially calcined talc crystals are used to make electrical insulating material such as talc ceramics, etc.

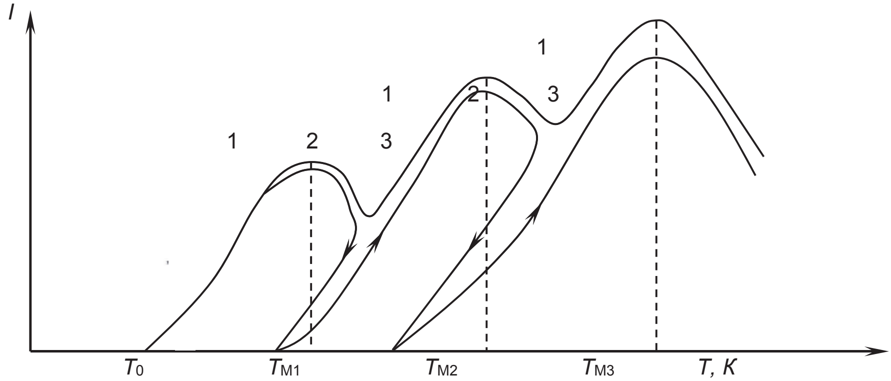


Fig. 5. Partial thermal cleaning method for DCT currents:
1 – linear heating; 2, 3 – partial thermal cleaning mode

Table 2. Activation Energy and Type of Calcium Sulfate Relaxers

No maximum	T_M, K	U_a, eV	Type Relaxers
1	92	0,06 ± 0,01	H^+
2	120	0,11 ± 0,01	H_3O^+
3	180	0,24 ± 0,02	H_2O Crystallization
4	209	0,36 ± 0,03	H_2O adsorbed
5	230	0,41 ± 0,04	OH, VL complexes
6	245	0,51 ± 0,06	H_3O^+
7	305	0,38 ± 0,04	H^+, H_3O^+, OH^-

Table 3. Activation energy and type of relaxers for $CuSO_4$ crystals·$5H_2O$

No maximum	T_M, K	U_a, eV	Type Relaxers
1	92	0,06 ± 0,01	H^+

2	121	0,11 ± 0,01	H_3O^+
3	175	0,23 ± 0,02	H_2O Crystallization
4	206	0,33 ± 0,03	H_2O adsorbed
5	230	0,42 ± 0,04	OH, VL complexes
6	248	0,48 ± 0,05	H_3O^+
7	290	0,34 ± 0,04	H^+, H_3O^+, OH^-

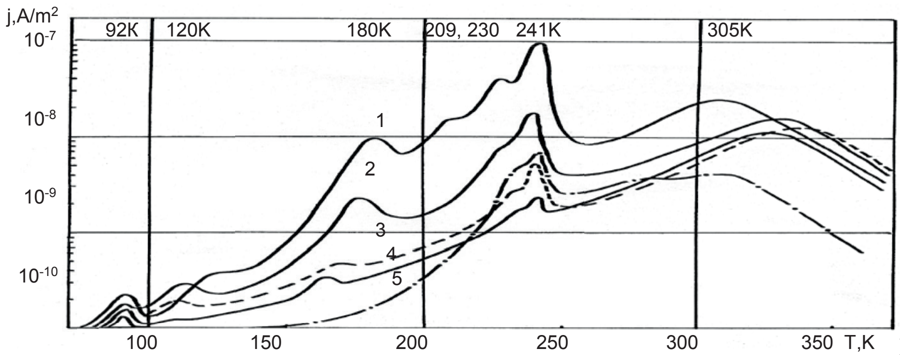


Fig. 6. Dependence of the density of calcium sulphate TCTD on the calcination temperature, T_n = 300 K: 1 – natural ($CaSO_4 \cdot \boldsymbol{2H_2}O$), 2 – T_{calc} = 120 ºC ($CaSO_4 \cdot \boldsymbol{H_2}O$), 3 – T_{calc} = 160 ºC ($CaSO_4 \cdot \boldsymbol{0.5H_2}O$), 4 – T_{calc} = 180 ºC ($CaSO_4$), 5 – 600 ºC

Fig. 9 shows the dependence of the density of the TCTS of magnesium hydrosilicate on the calcination temperature. For the density of the CTTS of crystals aged in HCl solutions, a slight increase in the maximum of 1 was observed, and the maximum of 6 shifted to the low temperature range. For calcined crystals, the maximum of 2 decreased in amplitude and shifted to the low temperature region. With an increase in the concentration of the HCl solution, this shift of maximum 6 increased, and the maximum value increased by more than an order of magnitude. After calcination, the reverse movement of maximum 6 was observed – to the high temperature region (Fig. 6–8). The amplitude of the maxima decreased significantly. The very low value of the activation energy of maximum 1 indicates that it is due to proton tunneling between oxygen ions (Ryzhkin et al., 2018; Lee et al., 2007).

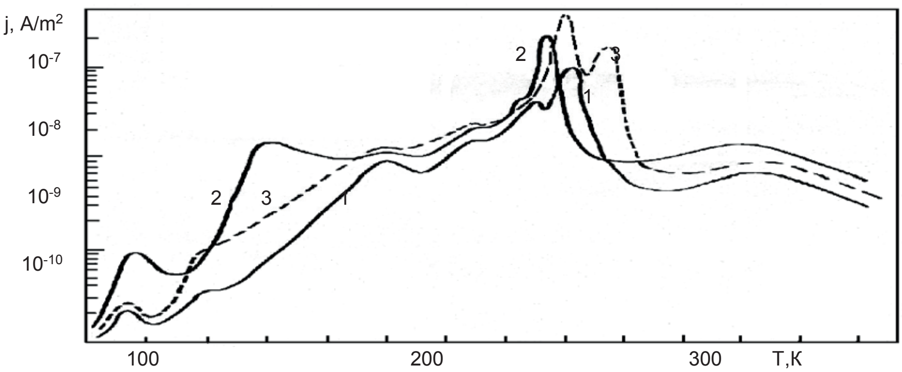


Fig. 7. Effect of calcium sulfate doping on the TSTD spectrum: 1 – natural, 2 – with an admixture of HCl, M(solution) = 7,4 mole/l (88 days), 3 – with an admixture of NH_4OH, M(solution) = 9,2 mole/l (95 days), $E_n = 2 \cdot 10^5$ V/m, T_n = 300 K

To determine the total depolarization charge, the area under the DCT current curve was integrated (i.e., the area under the curve is determined in the coordinates $I = f(T)$.

$$n = \int_{T_0}^{\infty} \frac{j(T)}{\beta d} dT \qquad (1)$$

where $\beta = dT/dt$ is the heating rate of the sample, d is the thickness of the sample.

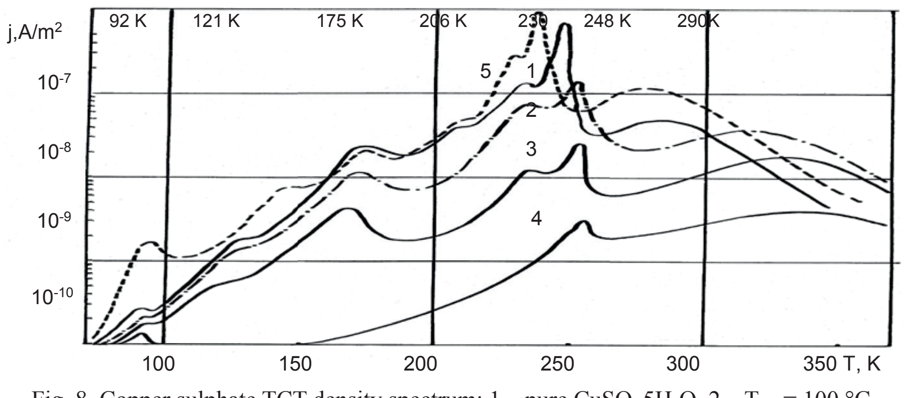


Fig. 8. Copper sulphate TCT density spectrum: 1 – pure $CuSO_4 \cdot 5H_2O$, 2 – T_{calc} = 100 °C, $CuSO_4 \cdot 3H_2O$, 3 – T_{calc} = 150°C, $CuSO_4 \cdot H_2O$, 4 – T_{calc} = 300 ºC, $CuSO_4$, 5 – $CuSO_4 \cdot 5H_2O$ [M(HCl) = $1 \cdot 10^{-5}$ mole/m^3]. $E_n = 2 \cdot 10^5$ V/m, T_n = 300 K, $\boldsymbol{\beta}$ = 5.5 K/min

Table 4. Parameters of magnesium hydrosilicate defects from TSTD spectra

No maximum	T_{max}, K	Energy Activation, U, eV	Concentration, n, m^{-3}	Type Relaxers
1	86	0,06 ± 0,01	$5{,}7 \cdot 10^{17}$	H^+
2	112	0,10 ± 0,01	$1{,}2 \cdot 10^{18}$	H_3O^+

3	180	0,19 ± 0,02	$3{,}5{\cdot}10^{19}$	H_2O crystallization
4	206	0,25 ± 0,02	$1{,}8{\cdot}10^{19}$	H_2O adsorbed
5	230	0,32 ± 0,04	$8{,}35{\cdot}10^{19}$	OH, VL complexes
6	251	0,41 ± 0,05	$5{,}4{\cdot}10^{20}$	H_3O^+
7	305	0,36 ± 0,04	$8{\cdot}10^{22}$	H^+, H_3O^+, OH^-

The low value of the activation energy and electrical conductivity of H_3O^+ defects ($\gamma = 8 \times 10^{-11}$ cm/m) is explained by the imposition of the tunneling effect of protons at low temperatures (Timokhin, 2014; Hibino, Mizutani, Yajima, 2000; Ryzhkin et al., 2018; Lee et al., 2007). Both single crystals and polycrystals were studied, and it was shown that the maximum No 2 of the TSTD spectrum and the low-temperature conductivity of polycrystals increased by 3–4 times compared to single crystals, that is, channels are formed in polycrystals that facilitate the transport of protons and the diffusion of oxonium groups H_3O^+. The wide seventh maximum at 305 K is explained by the volume charge, due, among other things, to the pressure of rocks in the place of occurrence of crystalline Materials. The influence of ultrasonic vibrations of the soil is also possible, which is noted in the work (Perez-Rodrigues et al., 2006).

In the presence of NH_4OH impurity, a different picture was observed: maximum 2 shifted to the low temperature region, and maximum 6 to the high temperature region (fig. 7, 8). At the same time, with an increase in the concentration of the impurity, the displacement of maximum 6 increased. For crystals with an impurity of NH_4OH, a significant increase and shift to the high temperature region of the maximum of 5 (more than 10 times) was observed, while other maxima increased approximately 2 times are shown in Table 2–4, where an unusually low activation energy value is observed for the maximum No 1. This was not observed for crystals with an admixture of HCl. It is obvious that for maximum 5 the main role is played by hydroxyl groups OH^-, and for maximum 6 the main role is played by oxonium ions H_3O^+. At the same time, in the presence of an impurity of NH_4OH and HCl, an increase and displacement of maximum 3 is practically not observed. Therefore, it can be assumed that this maximum is due to the relaxation of crystallization water molecules, which determines the number of water molecules in the formula unit of the crystal.

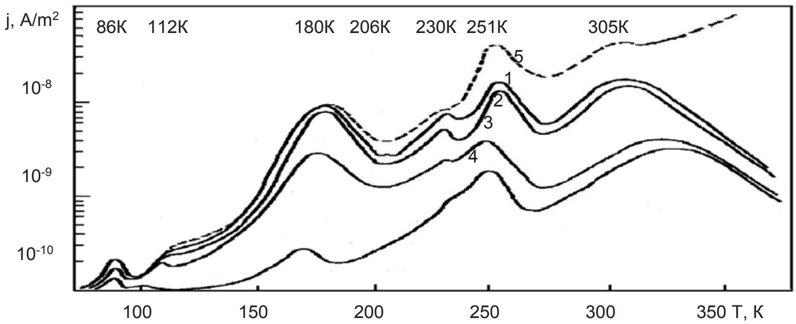


Fig. 9. Dependence of the density of TCTD of magnesium hydrosilicate on the calcination temperature at $E_n = 2 \cdot 10^5$ V/m, T_n=300 K: 1 – dried at T=300 K, 2 – calcined at T_{calc} = 573 K, 3 – T_{calc} = 1023 K, 4 – T_{calc} = 1323 K, 5 – natural, $E_n = 2 \cdot 10^5$ V/m

As can be seen from the above graphs, the maximum of 4 disappears after drying at 100°C, therefore, it is due to the relaxation of the adsorbed water molecules. The behavior of the maximum 7 corresponds to the relaxation of the bulk charge, that is, its value indicates the presence of various impurities in the air, including NH_4OH and HCl. That is, the TSTD spectra make it possible to control the number of water molecules in crystals. containing a different number of water molecules both in the structure and in the formula unit. For example, after calcination at appropriate temperatures (100°C, 120°C, 150°C), crystals of various structures can be obtained: $CuSO_4{\cdot}5H_2O$, $CuSO_4{\cdot}3H_2O$, $CuSO_4{\cdot}H_2O$, $CaSO_4{\cdot}2H_2O$, $CaSO_4{\cdot}0{,}5H_2O$.

Any crystal can be taken as a reference, for example, $CuSO_4{\cdot}5H_2O$ and other crystals can be compared with it. Natural crystals of biaqueous calcium sulfate were used. Measurements were carried out for both single crystals and polycrystals pressed from powder into tablets of the same thickness as single crystals. The number of water molecules in the formula unit and the purity of the studied crystals were controlled by X-ray phase analysis. The diffractogram showed the presence of a corresponding number of water molecules remaining in the crystal after calcination. For X-ray phase analysis, a DRON-UM1 X-ray unit with a cobalt anode was used. The wavelength of the K a line was 1,7902 Å. The calculation was made according to the Wolfe-Bragg formulas.

$$d = \frac{n\lambda}{2\sin(\theta/2)} \quad (2)$$

The data obtained (Table 5) coincide well with the data of the ASTM card index: $CaSO_4{\cdot}2H_2O$ map No 6-46, $CaSO_4{\cdot}0.5\,H_2O$ – card No 24-1067, $CuSO_4{\cdot}5\,H_2O$ – card No 11-646, $CuSO_4{\cdot}3\,H_2O$ – card No 22-249, $CuSO_4{\cdot}H_2O$ – map No 21-269, which indicates the high accuracy of the proposed method. Indices of a number of planes of the studied crystals were also determined. We have established that the ratio is fulfilled with great accuracy:

$$\frac{j_{m.refer}}{j_{m.x}} = \frac{N_{refer}}{N_x} \quad (3)$$

is true for both single crystals and polycrystals.

Table 5. Results of diffractogram calculation for crystal hydrates of calcium and copper sulfates

Experiment		ASTM		Experiment		ASTM	
$CaSO_4 \cdot 2H_2O$				$CuSO_4 \cdot 5H_2O$			
d, Å	I/I_o	d, Å	hkl	d, Å	I/I_o	d, Å	hkl
7,53	100	7,56	020	5,18	3	5,15	$11\bar{1}$
3,80	20	3,79	031	4,68	100	4,66	110
3,79	30	3,79	040	2,57	2	2,57	$22\bar{2}$
3,06	2	3,06	$11\bar{1}$	2,33	75	2,33	220
1,90	4	1,90	080				
1,89	4	1,90	062				
$CaSO_4 \cdot 0{,}5H_2O$				$CuSO_4 \cdot 3H_20$			
6,00	61	5,94	020,110	5,11	53	5,11	110
3,46	66	3,44	200	4,87	45	4,86	021
3,00	100	3,00	202,132	4,42	100	4,42	$11\bar{1}$
2,79	16	2,77	114,024	3,98	53	3,97	111
2,70	5	2,68	222,042	3,65	75	3,65	002
2,26	6	2,24	240,310	3,41	75	3,42	130
2,13	9	2,15	224	3,25	68	3,25	040
2,12	11	2,12	152,242	3,19	45	3,19	$11\bar{1}$
2,00	2	2,01	303,106	3,01	43	3,01	131
1,91	4	2,01	106	2,82	65	2,82	112
1,9	3	1,88	161	2,50	45	2,50	$22\bar{2}$
1,23	2	1,23	524				

Here $j_{m.refer}$, $j_{m.x}$ are the current densities of the third maximum of the TSTD, N_{refer}, N_x are the number of water molecules in the formula unit for the reference and the studied crystals. This ratio does not depend on the method of obtaining the crystal (natural or grown from solution). Table 6 summarizes the experimental results. Consequently, having a reference crystal with a known content of crystallization water molecules in the formula unit and a CTT spectrum, it is possible to determine the number of Nx water molecules in the formula unit of any crystal hydrate. There is no need to take the entire spectrum of CTTD, it is enough to obtain the third maximum and measure the maximum value of the CTT density of this maximum. Then, using formula (3), determine the number of crystallization water molecules in formula unit of the crystal hydrate under study.

As can be seen from Table 6, the error does not exceed (2... 5,8) %. This allows us to conclude that the ratio (3) holds for any pair of crystals, for example, for $CuSO_4 \cdot 3H_2O$ and $CaSO_4 \cdot 2H_2O$. In this case, $N_{refer} / N_x = 1.5$; the experiment yields $j_{refer} / j_x = 1.48$. The values j_m of maximum 3 for different crystal hydrates are linearly related, so it is necessary to determine the correlation of these values. The correlation coefficient was calculated using expression (4) (Rumer and Rybkin, 1977), where x_i, y_i are current values, $\bar{x}, \bar{y}$ are average values, N is the number of measurements.

Table 6. Ratio of the number of water molecules in formula units and TSTD densities for polycrystals and single crystals of crystal hydrates

Crystal Type	J_m, A/m^2	N_{refer} / N_x	j_{refer} / j_x	δ_0, %
Polycrystals				
$CuSO_4 \cdot 5H_2O$	$7{,}26 \cdot 10^{-9}$	1	1	-
$CuSO_4 \cdot 3H_2O$	$4{,}5 \cdot 10^{-9}$	5/3 = 1,66	1,62	2,4
$CuSO_4 \cdot H_2O$	$1{,}51 \cdot 10^{-9}$	5/1 = 5	4,8	4
$CaSO_4 \cdot 2H_2O$	$3{,}03 \cdot 10^{-9}$	5/2 = 2,5	2,4	4
$CaSO_4 \cdot 0{,}5H_2O$	$7{,}7 \cdot 10^{-10}$	5/0,5 = 10	9,45	5,8
Single crystals				
$CuSO_4 \cdot 5H_2O$	$2{,}1 \cdot 10^{-8}$	1	1	-
$CuSO_4 \cdot 3H_2O$	$1{,}18 \cdot 10^{-8}$	5/3 = 1,66	1,76	5,8
$CaSO_4 \cdot 2H_2O$	$8{,}8 \cdot 10^{-9}$	5/2 = 2,5	2,38	4,8
$CaSO_4 \cdot 0{,}5H_2O$	$2{,}25 \cdot 10^{-9}$	5/0, 5 = 10	9,55	4,5

The number of measurements was selected so that the probability of correlation of two uncorrelated values with $r > 0.8$ was less than 5%. For $N = 8$, it is 1–2%.

For example, let's consider the pair: $y = j_{refer}$ ($CuSO_4 \cdot 5H_2O$), $x = j_x$ ($CaSO_4 \cdot 2H_2O$). All the data for the calculation are summarized in Table 7, from where we get the value of the correlation coefficient r for several pairs of crystals, which turned out to be equal to 0.81.

$$r = \frac{\sum_{i=1}^{n} x_i y_i - N\bar{x}\,\bar{y}}{\left[\left(\sum_{i=1}^{n} x_i^2 - N\bar{x}^2\right)\left(\sum_{i=1}^{n} y_i^2 - N\bar{y}^2\right)\right]^{1/2}} \quad (4)$$

Table 7. Calculation of the correlation coefficient for the pair $CuSO_4 \cdot 5H_2O$ – $CaSO_4 \cdot 2H_2O$

№	x_i 10^{-8}, A/m^2	y_i 10^{-8}, A/m^2	$x_i^2 \cdot 10^{-16}$	$y_i^2 \cdot 10^{-16}$	$x_i y_i$, 10^{-16}
1	0,90	2,46	0,81	6,05	2,214
2	0,92	2,53	0,846	6,40	2,328
3	0,96	2,50	0,925	6,25	2,405
4	0,85	3,01	0,722	9,0	2,550
5	1,15	2,30	1,322	5,29	2,645
6	0,99	2,28	0,98	5,19	2,258
7	1,05	2,16	1,102	4,666	2,268
8	0,93	2,56	0,864	6,55	2,381

Conclusion. The proposed thermal activation diagnostics for determining the structure of crystalline materials of rocks can be used for other classes of crystal hydrates. Based on the results of the study of the TSTD spectra, the "Thermal Activation Diagnostics of the Structure of Crystalline Materials" was developed, which provides a complete analysis of the types and characteristics of structure defects in the study of calcined and doped crystals, as well as the concentration and number of molecules crystallization water in a formula unit, e.g., in crystals $CuSO_4 \cdot 5H_2O$, $CuSO_4 \cdot 3H_2O$, $CaSO_4 \cdot 2H_2O$, $CaSO_4 \cdot 0.5H_2O$ and others. The developed method can be used to analyze the deposit of crystalline materials and chemical cargoes upon arrival at the port of destination. With high humidity of rocks and during the transportation of dry materials, the number of crystallization water molecules in the formula unit can change, as a result of which the value of the material will significantly decrease to restore its chemical formula.

In addition, the absence of maximum No 3 in the TSTD spectrum indicates the absence of crystallization water molecules in the studied crystalline material, the absence of maximum No 4 indicates the absence of adsorbed water, the absence of maximum No 5 indicates the absence of OH ions, that is, its transformation into an anhydrous material, which is of great importance in the chemical and electrical industries when dehydrating crystals and powders from crystalline material. The presence of hydroxyl OH^- was shown by a significant increase in the maximum of 5 TCTD crystals doped with NH_4OH, and the presence of oxonium (maximum 6) indicates the presence of an acidic medium .

Consequently, the proposed diagnostics are especially important in the polar regions in mining enterprises and dry cargo ships transporting crystalline materials. A method for determining the number of water molecules in the formula unit for the reference and studied crystals using the method of thermally stimulated depolarization is proposed.The calculation of diffractograms for crystal hydrates with different numbers of water molecules in a formula unit has been carried out. Diagnostics of the quality of crystalline materials has been developed, which consists in changing the position and value of a number of TCTD maxima relative to the reference spectrum depending on the medium (for example, alkaline or acidic), which makes it possible to carry out environmental monitoring of mining deposits and the storage environment of crystalline materials (Modina, Khekert, Epikhin, 2021), as well as to control hydrogen leakage from storage cells and fuel tanks of mining vehicles (Modina, Khekert, Epikhin, 2021; Turkin et al., 2021a; Turkin et al., 2021b). The developed method for studying the structure of crystals is quite informative and environmentally friendly in comparison with X-ray diffraction and chemical analysis.

Thus, this diagnostics makes it possible to solve one of the fundamental scientific and technical problems of science and the national economy in the diagnosis of materials operating in extreme conditions, which will ensure an increase in personnel safety, as well as a decrease in the number of emergencies and accidents in industrial, transport and mining enterprises (Epikhin et al., 2024).

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