The polymorphic transition (PT) at \( \theta \approx 2^\circ \) in \( \text{C}_2\text{H}_4\text{O}_4 \) crystals was studied by experimental methods. The fact of PT in succinic acid (F. Dupré, La Tour, Ann. Phys. (1932) 18, 199) and the phase structures have long been established with different degrees of accuracy (\( \theta \)-phase) by J. S. Broady et al. Proc. Roy. Soc. (1959) A251, N 1267, 441; \( \alpha \)-phase G. D. Rieck, Rec. Trav. Chim. (1944) 63, n 4, 170), little is known about the transition itself. As objects served perfect monocrystals in \( \theta \)-phase grown from different solvents. A substantial hysteresis reaching tens of degrees was found. The reversibility of the process was first registered. The temperature of phase equilibrium was close to \( 116^\circ \text{C} \). The PT represents growth of daughter crystals (D) of the \( \alpha \)-phase inside the matrix crystal (M) which is analogous to growth of crystals from ordinary media. The morphology of growth of D inside M was studied. The anisotropy of the crystalline medium, steric hindrances and changes in specific volumes of the phases and due to strong hydrogen bonding highly diversify the course of the PT. Orientation of the phase crystals received primary emphasis. Measurements of 200 dim out directions of D in cross-ed nicols with respect to direction of cleavage \([001]\) in M gave statistical distribution in a sector between \( 0^\circ \) and \( 90^\circ \) close to an isotropic one. The diagram of the angles formed by straight-line boundaries of D inside M with respect to the same direction \([001]\) is constructed. Optical evidence is found correlating well with the diagram, thus indicating the orienting effect of M. While intersecting the twinning planes \([001]\) and \([101]\) of M, the straight-line boundaries of D maintain a fixed direction without "taking notice" of changes in orientation of the "building material". Using an extremely high hysteresis of PT \( \theta \approx 5^\circ \), we managed to indicate the boundaries in both lattices on the "hybrid" crystal at room temperature (Fig.1 a,b). Indicating was performed by the X-ray technique for monocrystals. It was found that the boundaries represent faces with low indices in the lattice of D and high indices in the lattice of M (except for the plane of cleavage). Based on the obtained evidence and the structural data, a molecular model of the boundary in \([010]\) of M is constructed.

**FIG.1 Micrograph of the PT in succinic acid (a) and the scheme of indicating of boundaries in the lattice of D (b).**

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**05. PHYSICAL PROPERTIES AND STRUCTURE**

**05.1-41 POLYMORPHIC TRANSITION IN SUCCINIC ACID CRYSTALS.** By N. N. Petropavlov, S. B. Vasil’chev, Institute of Chemical Physics of the USSR Acad. Sci., Pushchino, Moscow Region, USSR.

Though the fact of PT in succinic acid (F. Dupré, La Tour, Ann. Phys. (1932) 18, 199) and the phase structures have long been established with different degrees of accuracy (\( \theta \)-phase), the transition itself as objects served perfect monocrystals in \( \theta \)-phase grown from different solvents. A substantial hysteresis reaching tens of degrees was found. The reversibility of the process was first registered. The temperature of phase equilibrium was close to \( 116^\circ \text{C} \). The PT represents growth of daughter crystals (D) of the \( \alpha \)-phase inside the matrix crystal (M) which is analogous to growth of crystals from ordinary media. The morphology of growth of D inside M was studied. The anisotropy of the crystalline medium, steric hindrances and changes in specific volumes of the phases and due to strong hydrogen bonding highly diversify the course of the PT. Orientation of the phase crystals received primary emphasis. Measurements of 200 dim out directions of D in cross-ed nicols with respect to direction of cleavage \([001]\) in M gave statistical distribution in a sector between \( 0^\circ \) and \( 90^\circ \) close to an isotropic one. The diagram of the angles formed by straight-line boundaries of D inside M with respect to the same direction \([001]\) is constructed. Optical evidence is found correlating well with the diagram, thus indicating the orienting effect of M. While intersecting the twinning planes \([001]\) and \([101]\) of M, the straight-line boundaries of D maintain a fixed direction without "taking notice" of changes in orientation of the "building material". Using an extremely high hysteresis of PT \( \theta \approx 5^\circ \), we managed to indicate the boundaries in both lattices on the "hybrid" crystal at room temperature (Fig.1 a,b). Indicating was performed by the X-ray technique for monocrystals. It was found that the boundaries represent faces with low indices in the lattice of D and high indices in the lattice of M (except for the plane of cleavage). Based on the obtained evidence and the structural data, a molecular model of the boundary in \([010]\) of M is constructed.

**05.1-42 THE ORDER-DISORDER TRANSFORMATION IN THE \( \Pi \)-MOLECULAR COMPOUND PYRENE:PYROMELLITIC DIAHYDRIDE.** By A. M. Krayn, S. R. Meyh, and A. E. Samson, Noyes Laboratory of Chemical Physics, California Institute of Technology, Pasadena, CA 91125, U. S. A.

The crystal structure of pyrene:pyromellitic dianhydride (PMDA) has been determined at temperatures of 295, 280, 185, 180, 100 and 19 K using the low-temperature accessory designed by the author (J. Appl. Cryst. 13, 495 (1980)) for a four-circle diffractometer. Earlier results for the ordered and disordered structures (Herbstel & Snyman, Phil. Trans. Roy. Soc. London A264, 355 (1969)) are confirmed and extended. At 295 K \( \Delta H = 13.94(1), \; B = 9.34(1), \; c = 7.31(1), \; \beta = 93.65(1)^\circ \), space group \( P2_1/n, \; Z = 4, \; a = 13.664(3), \; b = 9.852(2), \; c = 14.260(5), \; \beta = 95.80(2)^\circ \), space group \( P2_1/a, \; Z = 4, \; a = 19.36(1), \; b = 9.85(2), \; c = 13.36(1) \), space group \( I2_1/a, \; Z = 1, \; a = 14.15(1), \; b = 9.37(1), \; c = 7.25(1) \), space group \( P2_1/a, \; Z = 4, \; a = 13.664(3), \; b = 9.852(2), \; c = 14.260(5), \; \beta = 95.80(2)^\circ \), space group \( P2_1/a, \; Z = 4, \; a = 19.36(1), \; b = 9.85(2), \; c = 13.36(1) \), space group \( I2_1/a, \; Z = 1, \; a = 14.15(1), \; b = 9.37(1), \; c = 7.25(1) \), space group \( P2_1/a, \; Z = 4, \; a = 13.664(3), \; b = 9.852(2), \; c = 14.260(5), \; \beta = 95.80(2)^\circ \), space group \( P2_1/a, \; Z = 4, \; a = 19.36(1), \; b = 9.85(2), \; c = 13.36(1) \), space group \( I2_1/a, \; Z = 1, \; a = 14.15(1), \; b = 9.37(1), \; c = 7.25(1) \). Based on the transition temperature the "two independent pyrenes are separated by a rotation about the normal to the molecular plane at 19 K this angle is 12.6° and it decreases slowly with rising temperature. Above the transition temperature the pyrene molecules have a "resonance" structure. The diagram of the angles formed by straight-line boundaries of D inside M with respect to the same direction \([001]\) is constructed. Optical evidence is found correlating well with the diagram, thus indicating the orienting effect of M. While intersecting the twinning planes \([001]\) and \([101]\) of M, the straight-line boundaries of D maintain a fixed direction without "taking notice" of changes in orientation of the "building material". Using an extremely high hysteresis of PT \( \theta \approx 5^\circ \), we managed to indicate the boundaries in both lattices on the "hybrid" crystal at room temperature (Fig.1 a,b). Indicating was performed by the X-ray technique for monocrystals. It was found that the boundaries represent faces with low indices in the lattice of D and high indices in the lattice of M (except for the plane of cleavage). Based on the obtained evidence and the structural data, a molecular model of the boundary in \([010]\) of M is constructed.


The different states of order of hexadecane \((\Pi 0314)\) included in channels formed by urea along the hexagonal c-axis have been investigated. At 150 K a continuous transition from a hexagonal high-temperature phase to an orthorhombic (distorted pseudo-hexagonal) structure takes place preceded by a sharpening of diffuse intensity (streaks) at the locations of the rhombohedral superstructure reflections. This diffuse intensity was shown to be mainly of elastic or quasi-elastic origin by a measurement on the 3-axis neutron diffractometer D10 at ILL in "elastic" and energy-integrated configuration. The transition is connected with a lateral ordering of the adducts, the superstructure being referred to an interaction between chain and channel. Hexadecane, which has a length close to but not equal to 2c of urea shows sharp reflections with \( c = c/2 \) and \( a^* = a^* \) of urea as well as diffuse layers perpendicular to \( c^* \) corresponding to its own chain-length. Below 119 K, x-ray reflections on these layers occur belonging to a lattice which has a and b of urea and is commensurate with higher order in c. With decreasing temperature these new reflections grow at the expense of the \( c \)-wave until they finally prevail indicating an ordering of the chains according to their length. There is a hysteresis of about 15 K. At the same time first and higher order satellites are found due to a longitudinal modulation of the structure along c caused by a "resonance" between guest and host.