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Structural deformations and phase transitions of normal paraffins

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Rotation of particles (atoms or molecules) around a point or an axis is a specificity of rotator crystals. Normal paraffins n-C_nH_{2n+2} are classical representatives of rotator substances. This makes available to take them as examples for investigation of the rotator-crystal state, which is among the less investigated phase states of the matter. Transformation of a substance into a rotator-crystal state can be caused for instance by a heating and related to a change of the type of thermal movement of particles (atoms, molecules) due to the loss of the fixed orientation in the structure. In the case of n-paraffins, chain molecules acquire an ability of oscillator-rotator thermal motions around their axes.

Structural deformations and polymorph transitions were investigated with using of high temperature X-ray powder diffraction method (the temperature step is 0.1–0.5ºC) for odd n-paraffins (*orthorhombic*) and even ones (*triclinic* and *monoclinic*) in the range of n = 17–36 (homological purity 97–99 %).

All the n-paraffins except of n = 18 transform into the low-temperature rotator-crystal state *rot.*1. Short-chain n-paraffins (n = 17–28) transform into the orthorhombic rotator-crystal phase $Or_{rot.1}$ [1, 2] and long-chain n-paraffins (n = 29–36) transform into the triclinic rotator-crystal phase $Tc_{rot.1}$ [3, 4]. The transformations of long-chain n-paraffins of n = 27 and 30 into the phases $Or_{rot.1}$ and $Tc_{rot.1}$ respectively run through the intermediate monoclinic rotator-crystal phase $M_{rot.1}$. The transformation of n-paraffins of n = 33–36 into the phase $Tc_{rot.1}$ runs through the intermediate triclinic crystal phase Tc_{cryst}. Only the "middle" (triclinic and orthorhombic) members of the n-paraffin homological members (n = 22–26) transform into high-temperature rotator-crystal state *rot.*2 (hexagonal phase $H_{rot.2}$). Cooling melts displayed the temperature reversibility and unreversibility of short-chain (n = 17–28) and long-chain (n = 29–36) n-paraffin transformations respectively.

The variety of the rotator crystal types (rotator-crystal states) increases owing to the crystal lattice shows the signs of both dynamic and static models as well as due to different molecules possess different character of thermal movement [1]. High temperature phase transition in long-chain n-paraffins are accompanyed by lowering of crystal structure symmetry: $Or_{cryst} \rightarrow M_{rot.1}$ (n = 27), $Or_{cryst} \rightarrow Tc_{rot.1}$ (n = 29 and 31), $M_{cryst} \rightarrow Tc_{rot.1}$ (n = 32), $Or_{cryst} \rightarrow Tc_{cryst} \rightarrow Tc_{rot.1}$ (n = 33 and 35), and $M_{cryst} \rightarrow Tc_{cryst} \rightarrow Tc_{rot.1}$ (n = 34 and 36). The symmetry lowering proceeds due to a re-packing molecules in the n-paraffin structure. Under certain conditions, less symmetric but more dense packing can be formed.

Rotator phases possess some physical features, which are not typical for crystal substances. They are of a pronounced plasticity, which is the most important operating characteristic of n-paraffins. Eight binary phase diagrams were plotted using data for mixtures of n-paraffins.

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Ab initio direct solution from powder data lower than atomic resolution

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Direct methods work best when large numbers of well-determined reflection intensities have been collected to atomic resolution (better than 1.2Å). For a powder diffraction experiment, this situation is rarely the case. Powder diffraction patterns generally contain contributions from many overlapped reflections meaning that the condition of 'well determined' reflection intensities is not met. For moderately sized crystal structures, even with powder diffractometers of the highest angular resolution, it is impossible to obtain individual integrated intensities at atomic structural resolution.

The dual-space-based *Shake-and-Bake* procedure is one of the most successful direct methods for phasing single crystal diffraction data. A new method, termed Powder *Shake-and-Bake* [1] and implemented in the computer program *PowSnB*, addresses the handling of multiply overlapped reflections and the extension of powder diffraction data to atomic resolution *via* empirical estimation of the integrated intensities. *PowSnB* performs in each cycle of *SnB* iteration (i) a re-partitioning of overlapped-reflections (*via* partial structural information from the previous cycle), (ii) a reciprocal-space phase refinement (*via* the reduction of the values of a statistical minimal function), and (iii) a real-space density modification (*via* peak picking).

Successful *PowSnB* applications to experimental powder diffraction data lower than atomic resolution have demonstrated the power of the powder *Shake-and-Bake* method. This research was partially supported by a Knowledge Building grant from ExxonMobil Research and Engineering.

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Waste-free synthesis of the metallodrug bismuth subsalicylate

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