

s9.m1.p5 **Low temperature structure and dynamics of β -chloropivalic acid.** C. Buhrmester, H. Fuess, *TU Darmstadt, Petersenstr. 23, D-64287 Darmstadt, Germany*
 Keywords: molecular interactions, organic materials, molecular solids.

Prominent members of ordered-disordered phase transitions are neopentane¹ and pivalic acid². The shape of neopentane (CH₃)₄C can be regarded as a sphere, where as pivalic acid CH₂Cl(CH₃)₂CCOOH forms dimers and therefore is cigar-like shaped. The surface of these bodies are smooth and the rotation is not sterically hindered.

The dynamical rotation of the cigar-like shaped compounds is mostly uniaxial, e.g. the molecule rotates around the long axis of the dimer unit.

By substitution of one CH₃-group with a bulky or/and heavy group, the order-disorder decreases or may even disappears.

The substitution of a CH₃-group with CH₂Cl in pivalic acid leads to β -chloropivalic acid (3-chloro-2,2-dimethylpropionic acid). The phase transitions and the structures in the different phases have been investigated³.

Three different phases were established by thermal analysis. An indication of a fourth phase is given by second moment ¹H NMR measurement between T_{IV→III}=80-100K. In this presentation we will concentrate on the low temperature phases of β -chloropivalic acid CH₂Cl(CH₃)₂CCOOH.

The single crystal analyses of phases III and IV phases do not show significant structural changes. Powder patterns up to the phase transition III→II show additional reflections.

¹H NMR measurements show the dynamical motion in the low temperature phases, revealing the rotation of the CH₃-groups and CH₂Cl-group at 80K.

s9.m1.p6 **The Surprising Polymorph C of Cimetidine: synchrotron radiation to the rescue.** H. Birkedal[#], A. Bauer-Brandl^{*}, P. Pattison[#], [#]*Institute of Crystallography, BSP, University of Lausanne, CH-1015 Lausanne, Switzerland*, ^{*}*Institute of Pharmacy, University of Tromsø, Universitetsvn. 57, N-9037 Tromsø, Norway*.
 Keywords: polymorphism, cimetidine, synchrotron radiation.

Cimetidine, C₁₀H₁₆N₆S, is an anti-ulcer drug that functions as an H₂ histamine receptor antagonist. Till now, four polymorphic forms have been found¹. The crystal structures have been determined for the A² and D³ form. Crystals of polymorphs B and C are too small to be measurable with a home source.

We solved the structure of the C form using synchrotron radiation data collected on a crystal measuring 16×16×80 μm^3 . Surprisingly, the unit cell is very large and anisotropic in shape: $a = 81.226(16)$, $b = 4.900(1)$, $c = 19.180(4)$ Å, $\beta = 100.72(3)^\circ$ at RT. The space group is C2/c indicating that Z' = 3. The unit cell suggests an extended conformation of the molecules which is confirmed by the refined structure. The three independent molecules have almost the same geometry. The extended geometry is at odds with the two previously known polymorphs, that both adopt folded conformations.

High resolution synchrotron powder diffraction confirms that the large unit cell is present throughout the bulk material.

Very recently, a folded molecular structure was proposed for the C polymorph based on solid-state NMR data⁴. The NMR conformation is in complete disagreement with the present structural results. The NMR data show that only one conformer is present. This can be understood by the observation that the three independent molecules have almost the same conformation. Possibly it is the fact that Z' > 1 that poses problems in the interpretation of the NMR data.

Finally, the relative stability of the conformers found in the A, D and C polymorphs is investigated by *ab initio* calculations. These reveal that the A conformer is the more stable followed by D and C.

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