MS37-P4 Understanding phase transformation behaviour in aliphatic amino acids

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Polymorphism in DL-methionine and DL-norleucine was reported in the early 1950's in pioneer X-ray studies by Mathieson[1] and since then it has been subject to many detailed structural, thermoanalytical and spectroscopic studies. Amino acids with aliphatic side chains are particularly suitable for systematic study as they form sets of highly related structures which contain well-defined hydrogen bonded bilayers. The bilayers arrange to form structures where the hydrophobic side chains interact with each other through van der Waals interactions only. They exhibit remarkable thermal behaviour. Phase transformations in these materials are fully reversible and involve concerted molecular displacements and can therefore exhibit several forms, but their presence may be elusive and extremely hard to record. Even small degrees of rotation about bonds in the aliphatic chain can result in the formation of new phases. In a previous study[2], we presented the crystal structure of the elusive high temperature form of DL-norleucine. This material undergoes two single crystal to single crystal transformations leading to three unique forms at different temperatures. We showed that concerted translation of each bilayer in the crystal led to a sequence of $C2/c$>$P2_1/c$>$C2/c$ transformations. It has been shown that related amino acids (DL-norvaline & DL-aminobutaric acid) exhibit similar transformation patterns[3,4]. The low temperature phase of DL-norvaline has not been successfully determined, but it can be assumed that it should be $C2/c$. In fact, recently re-determined high and low temperature phases of DL-aminobutaric acid are shown to be a mixture of $P2_1/c$ and $C2/c$ forms[5]. To understand the driving force behind these phase transition phenomena we have extended our studies to other aliphatic amino-acids. Our recent X-ray diffraction studies, complemented by DSC and Hot-stage Microscopy, on 2-aminoheptanoic acid ($C_7H_{15}NO_2$) show complex thermal behaviour over a wider temperature range. We examined the 30K - 400K range and found five possible forms. Three of these forms follow fully reversible $C2/c$>$P2_1/c$ >$C2/c$ transformations. The remaining two low temperature forms are even more complicated and are currently under investigation.

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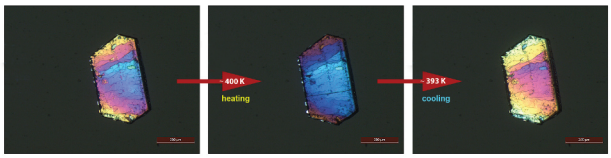

Figure 1. Hot-stage polarised optical microscopy of the highly reversible transformation of 2-aminobutaric acid in the range of 393K to 400 K.

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