MS38. Combining crystallographic information with other methods

Chairs: Marco Milanesio, Poul Norby

MS38-P1 Polymorph screening and crystal structure solution of 3-methylglutaric acid

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In solid-state NMR, as in almost every analytic technique, standard samples are needed to calibrate equipment in order to validate routine data collection. 3-Methylglutaric acid is a potential reference substance even though its crystal structure is unknown. [1]

As the crystal structure can influence the solid state NMR spectrum, the occurance of polymorphs under the usual experimental conditions has to be investigated.

3-Methylglutaric acid crystallizes readily from a variety of solvents. A representative set of commonly used solvents was selected and the crystallisation performed at room temperature and at elevated temperature. To exclude phase changes at higher temperatures DTA-TG was employed. As no diverging phases were identified by X-ray powder diffraction, the structure was determined by single crystal X-ray diffraction.

To obtain data matching the experimental conditions of solid-state NMR, the diffraction measurement was carried out at -100 °C as well as at 20 °C. The determined structures were identical within the thermal expansion as expected, similar to the results of earlier executed differential thermal analysis.

3-Methylglutaric acid crystallises in the space group $P 2_1/c$ with four molecules per unit cell (general position) and the lattice parameters

$a = 13.849$, $b = 5.323$, $c = 10.128$ and $\beta = 110.284$ ($R = 7.73$) at -100 °C and

$a = 13.909$, $b = 5.367$, $c = 10.307$ and $\beta = 110.555$ ($R = 5.29$) at room temperature (Fig. 1).

[1] D. H. Barich *et al.*, *Solid State Nucl. Magn. Reson.* 2006, *30*, 125-129.

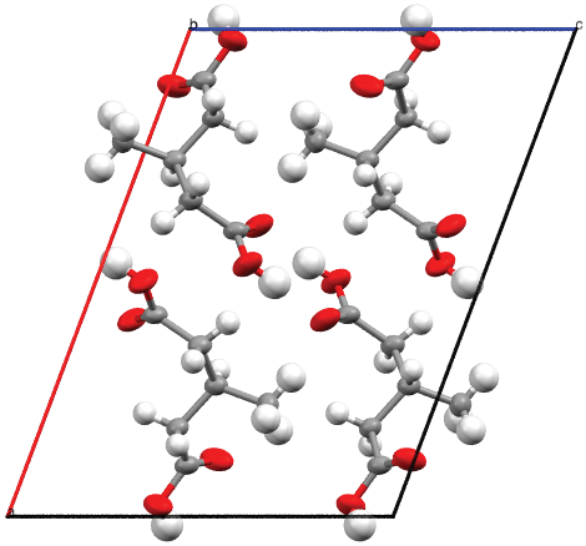

Figure 1. View along b axis (at room temperature).

Keywords: structure determination, NMR, organic compounds, polymorph screening