

## General Remarks

All fine chemicals were purchased from Aldrich and used as it is. Solvents were used as received when experiments were conducted in air. Flash chromatography was performed on 100-200 mesh silica gel.  $^1\text{H}$ - and  $^{13}\text{C}$ - Nuclear Magnetic Resonance (NMR) spectra were recorded on JEOL-400 MHz spectrometers at ambient temperature in  $\text{CDCl}_3$  which were purchased from Sigma Aldrich. Chemical shifts (ppm) are referenced to the residual solvent peak. Coupling constants,  $J$ , are given in hertz. Abbreviations used in the designation of the signals: s = singlet, d = doublet, dd = doublet of doublets, ddd = doublet of doublet of doublets, dt = doublet of triplets, t = triplet, td = triplet of doublets, m = multiplet. X-ray crystallographic analysis was collected on a Rigaku Oxford Diffraction Supernova diffractometer using  $\text{Cu K}\alpha$  radiation.

## X-ray Structure Determinations Technical Protocol

The crystals of **3**, and **4** were immersed in cryo-oil, mounted in a loop, and measured at a temperature of 120 K or 170 K. The X-ray diffraction data of **3** was collected a Rigaku Oxford Diffraction Supernova diffractometer using  $\text{Mo K}\alpha$  radiation. The X-ray diffraction data of **4** was collected on a on a Bruker Kappa Apex II diffractometer using  $\text{Mo K}\alpha$  radiation. The *CrysAlisPro* [S1] software package was used for cell refinements and data reductions for **3** and the *Denzo-Scalepack* [S2] software package for **4**. A multi-scan absorption correction (*CrysAlisPro* [S1] for **3** and *SADABS* [S3] for **4**) was applied to the intensities before structure solutions. Structures were solved by intrinsic phasing (*SHELXT* [S4]) method. Structural refinements were carried out using *SHELXL* [S5] software with *SHELXLE* [S6] graphical user interface. Hydrogen atoms were positioned geometrically and constrained to ride on their parent atoms, with  $\text{C-H} = 0.95 - 0.98 \text{ \AA}$  and  $U_{\text{iso}} = 1.2\text{-}1.5 U_{\text{eq}}$  (parent atom).

## References

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