Electronic Supplementary Material (ESI) for CrystEngComm.

Isotopomeric polymorphism in a "doubly-polymorphic" multi-component molecular crystal

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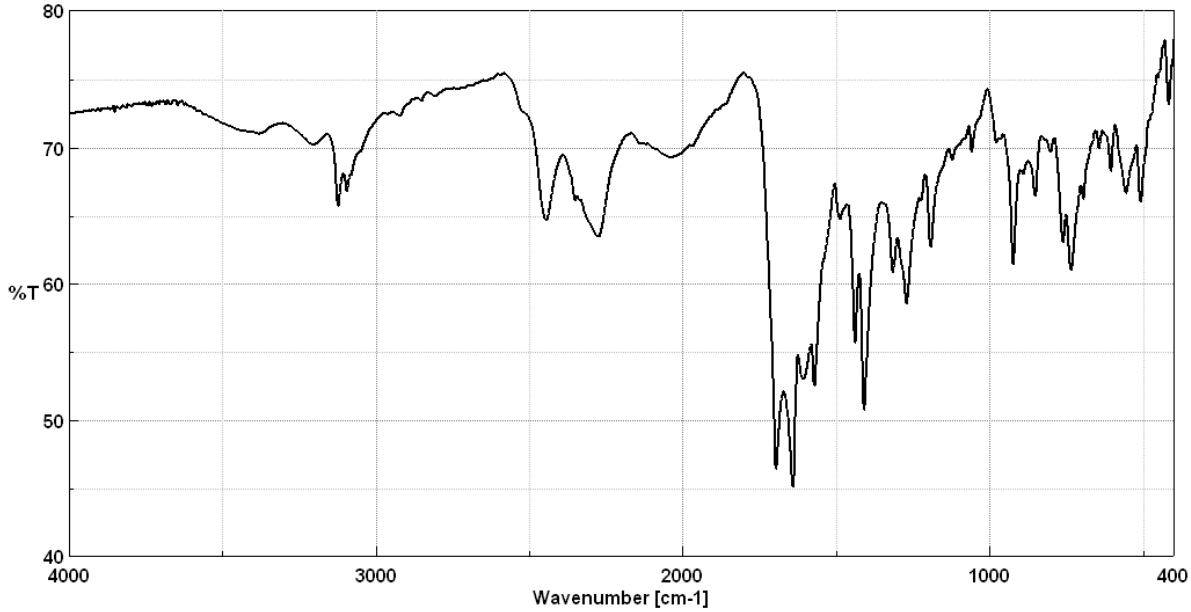
ESI

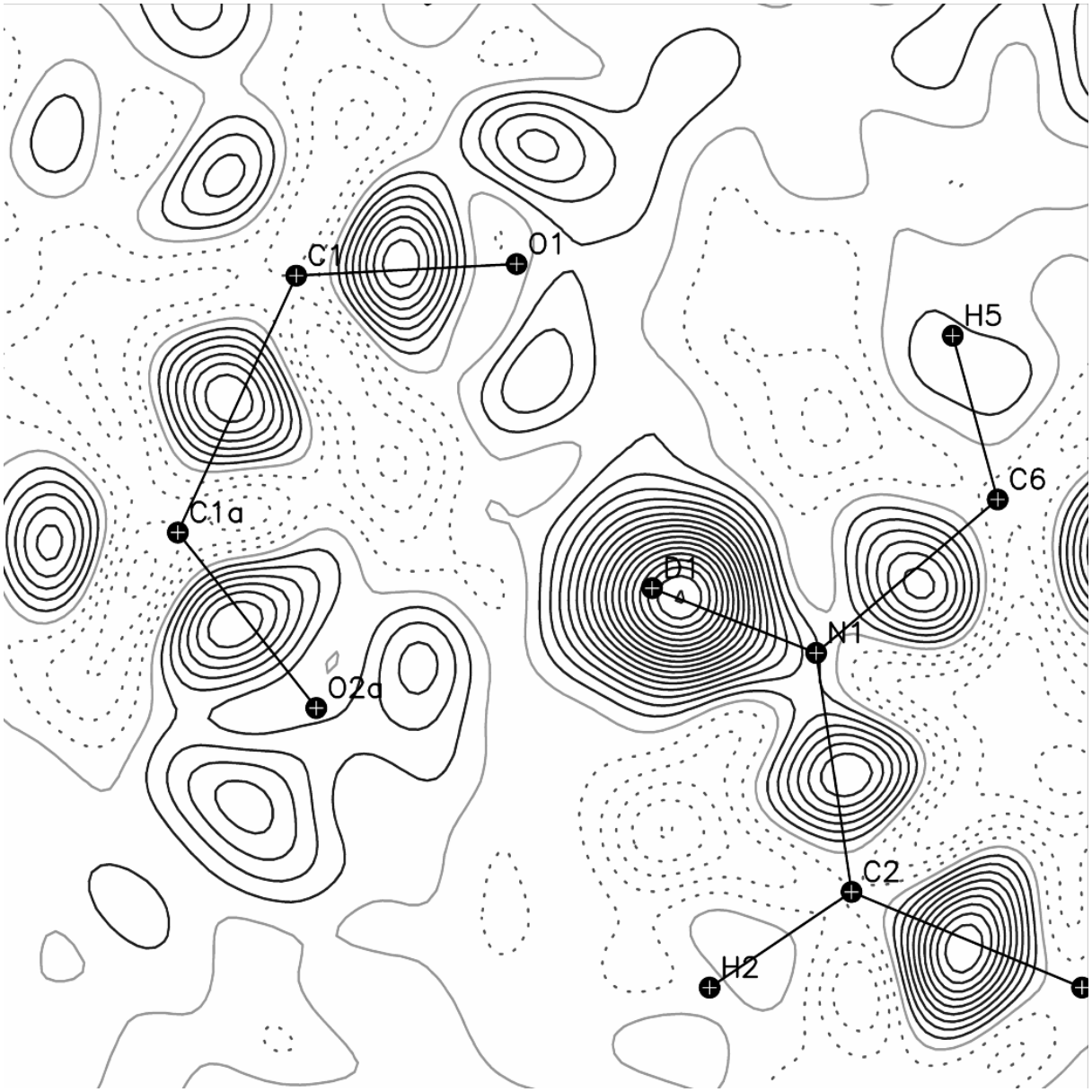
Figure ESI_1 – IR spectrum of IN_2–d-OA Form I, showing absence of N–H stretch



IR spectrum (KBr) of IN_2–d-OA Form I. 3123 + 3095 (w) C–H stretch; 2445 + 2277 (m) N–D stretch; 2040 (br) N–D···O HB; 1696 (s) C=O amide stretch; 1640 (s) CO_2^- asymmetric stretch; 1607 + 1572 (w) N–D bend; 1439 (s) CO_2^- symmetric stretch; 1409 (s) C–N stretch

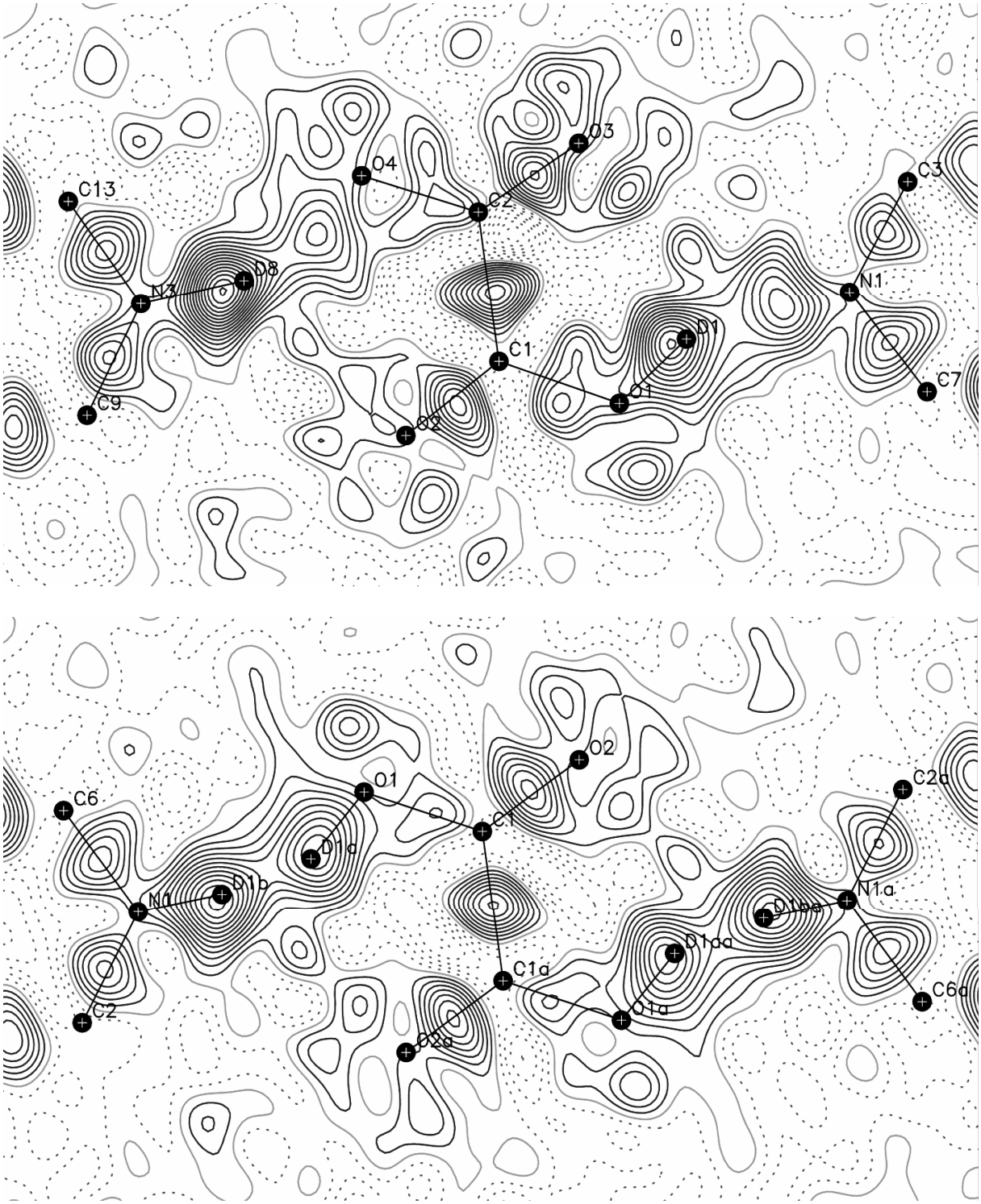
Fourier difference maps for IN_2–d-OA Forms I and II

Form I



Difference Fourier map in the O1–N1–O2a plane of IN_2–d-OA Form I; $\sin\theta/\lambda < 0.78$ Å^{-1}, clearly showing D1 to be transferred from the oxalic acid moiety.

Form II



Difference Fourier maps of IN_2–d-OA Form II in the OA plane after refinement in the (correct) supercell (top) and the (incorrect) small cell (bottom); $\sin\theta/\lambda < 0.78$ Å^{-1}. The correct supercell model shows that one D atom (D8) is fully transferred to the isonicotinamide moiety, while the other (D1) is retained by the oxalic acid; the lone pair density on N1 is also evident in this map. The incorrect 50:50 disorder model for the D1 atoms in the lower, small cell, map was forced by the wrong choice of cell, and led to poor modelling of the heavier atoms (evidenced by distorted ADPs; see Figure 5 of MS).

Bond lengths in IN_2–d-OA Forms I and II

			C~O / Å		∠CNC / °
Form I		C1–O1	1.2468(8)	C2–N1–C6	122.40(6)
		C1–O2	1.2626(8)		
Form II	supercell	C1–O1	1.3056(14)	C3–N1–C7	117.65(10)
		C1–O2	1.2181(14)		
		C2–O3	1.2463(14)	C9–N3–C13	122.12(10)
		C2–O4	1.2506(14)		

C–O bond lengths and CNC bond angles in both polymorphic forms of IN_2–d-OA