

Supplementary Materials: Multicomponent Solids of DL-2-Hydroxy-2-Phenylacetic Acid and Pyridinecarboxamides

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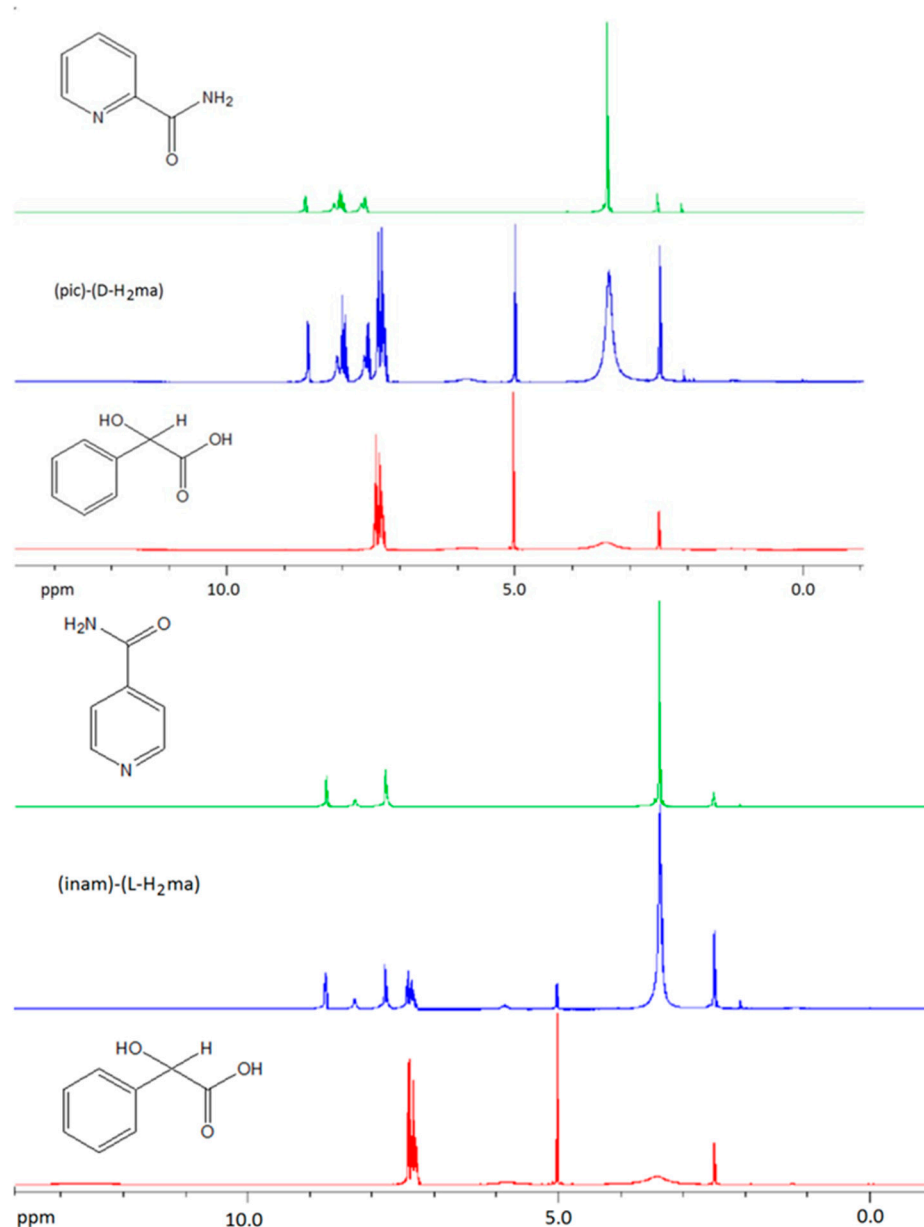


Figure S1. ¹H NMR spectra in DMSO-d₆ of cococrystals **1** (upper) and **3** (lower) comparative with their coformers.

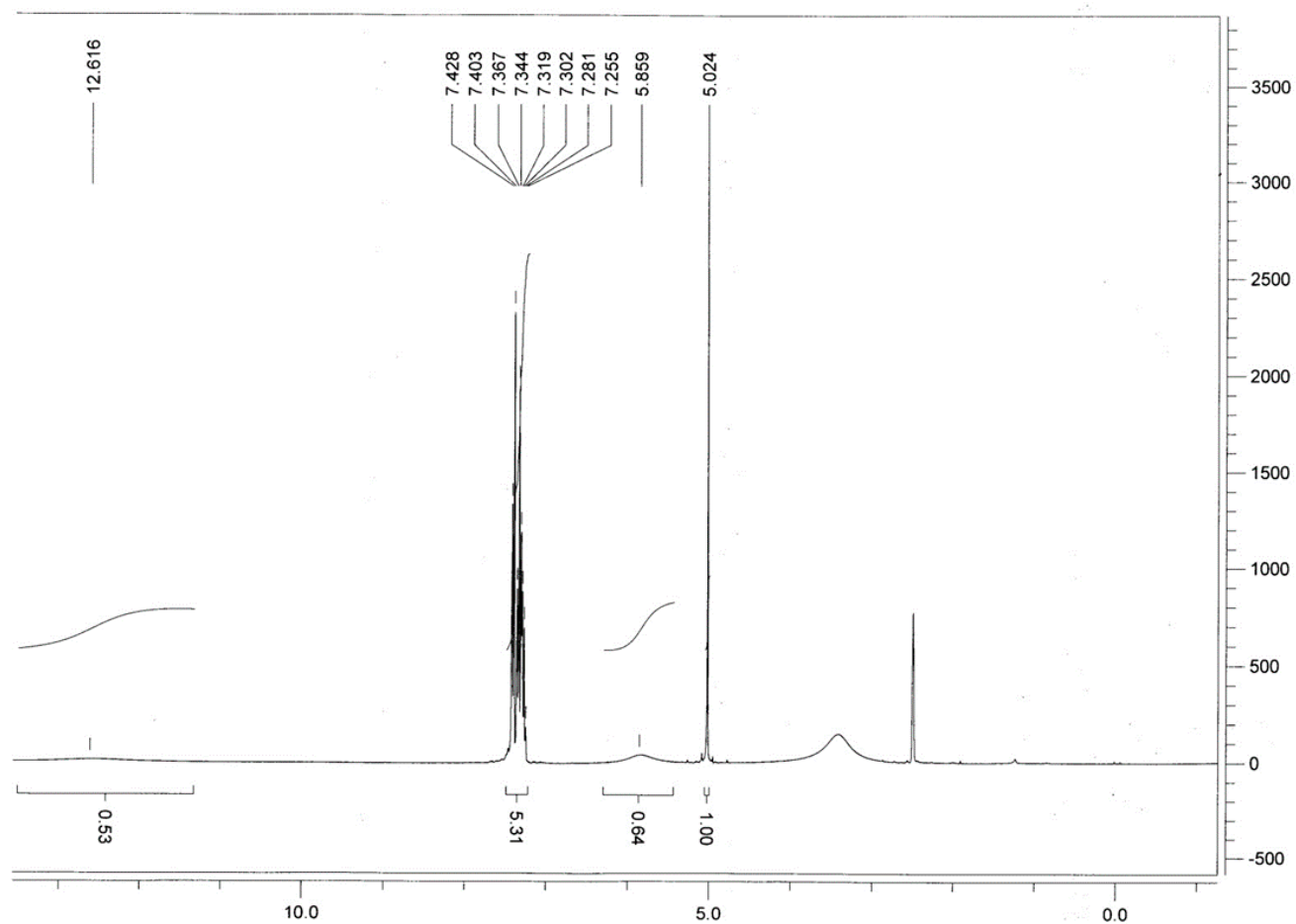


Figure S2. ^1H NMR spectrum of DL-H₂ma.

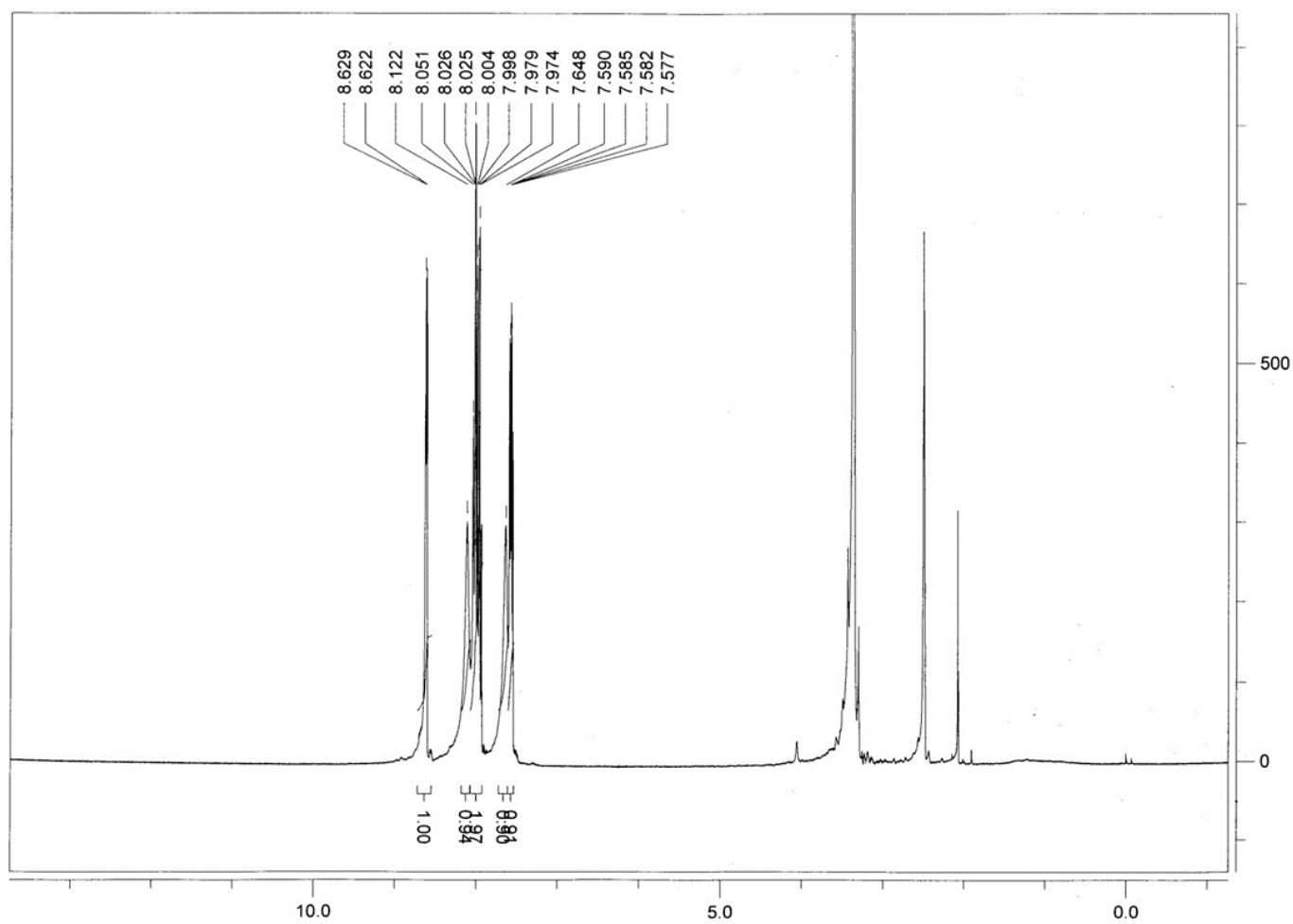


Figure S3. ^1H NMR spectrum of **pic**.

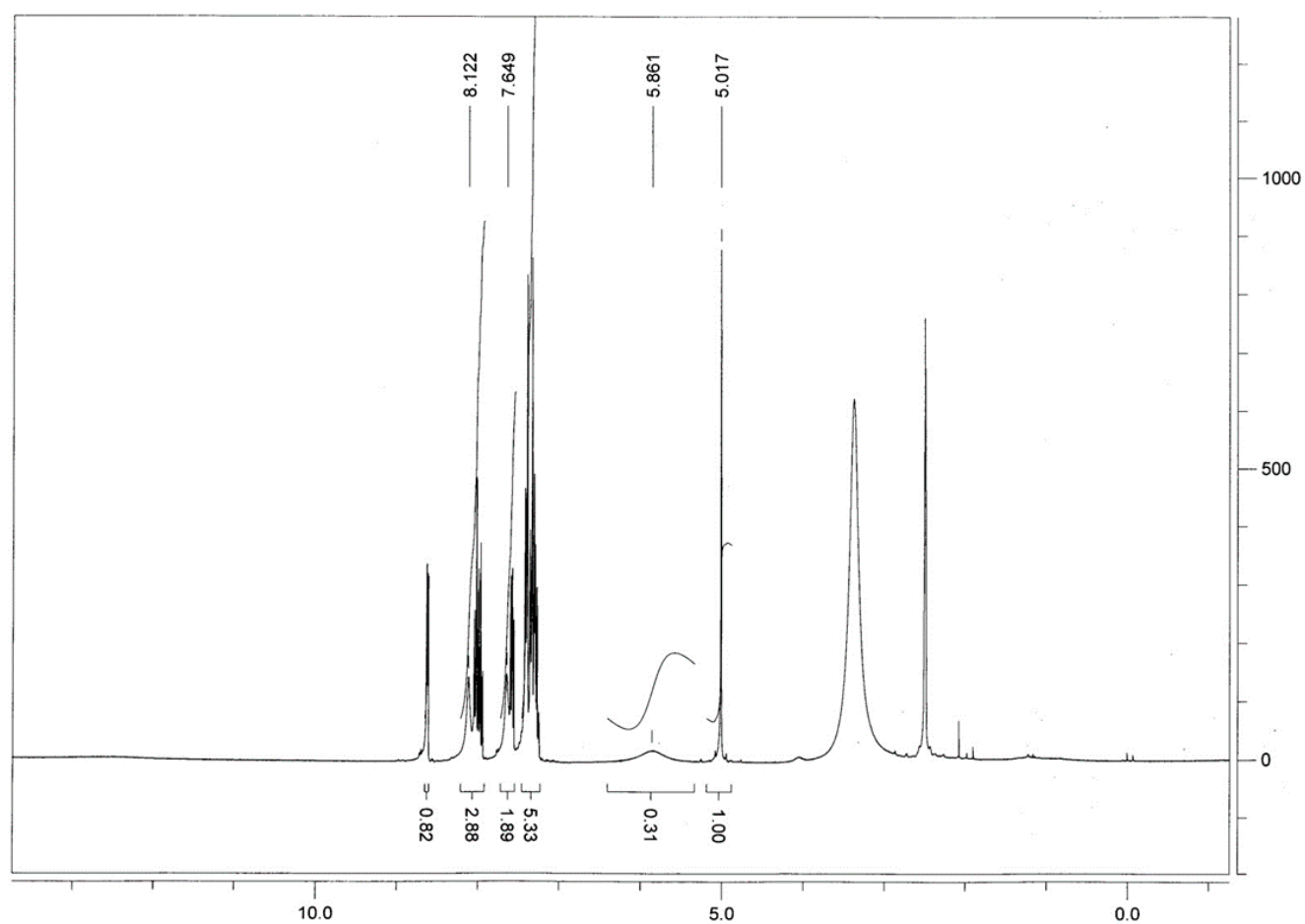


Figure S4. ^1H NMR spectrum of (pic)-(D-Hzma) (1).

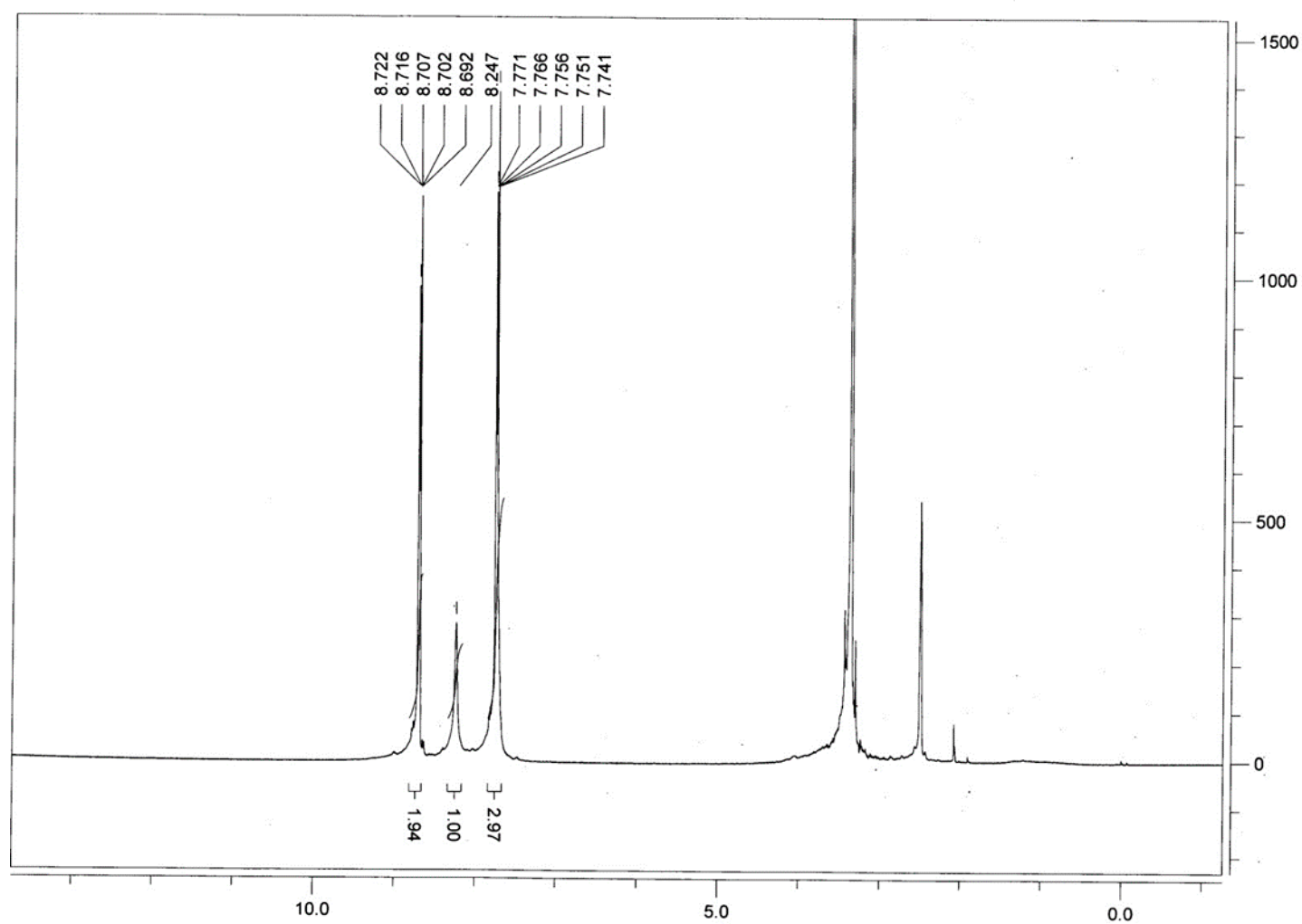


Figure S5. ^1H NMR spectrum of *inam*.

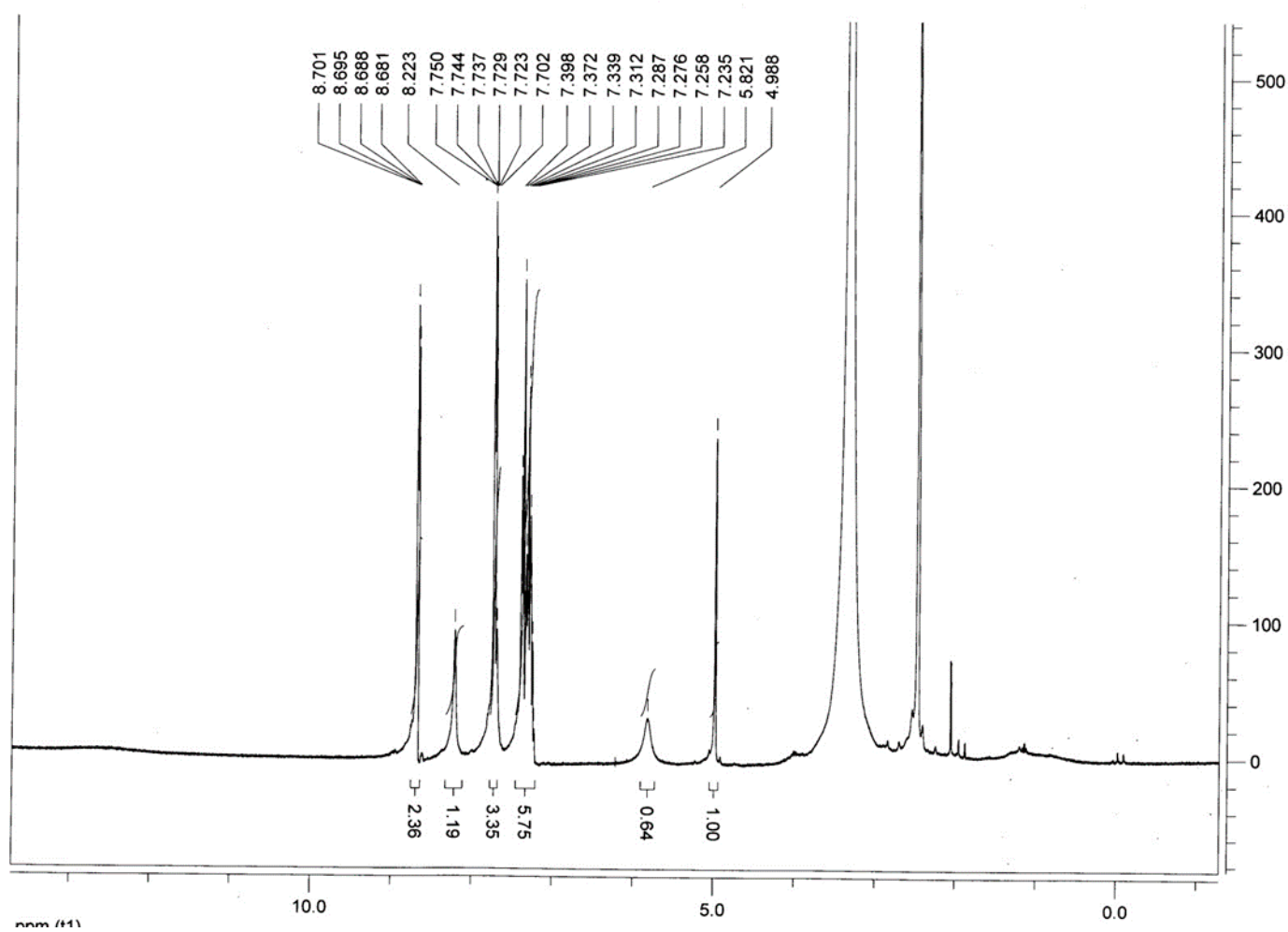


Figure S6. ^1H NMR spectrum of (inam)-(L-Hzma) (3).

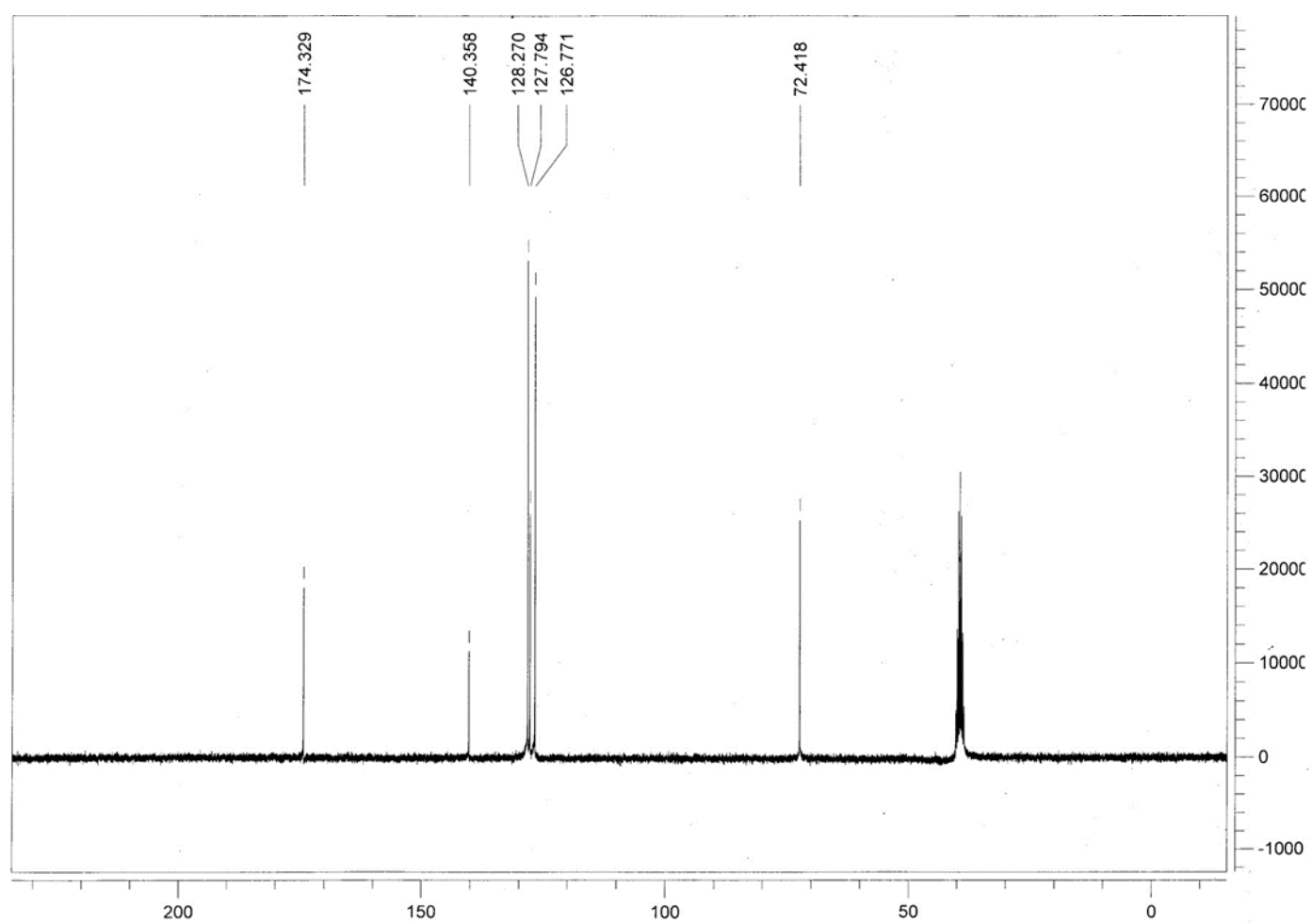


Figure S7. ^{13}C NMR spectrum of DL-H₂ma.

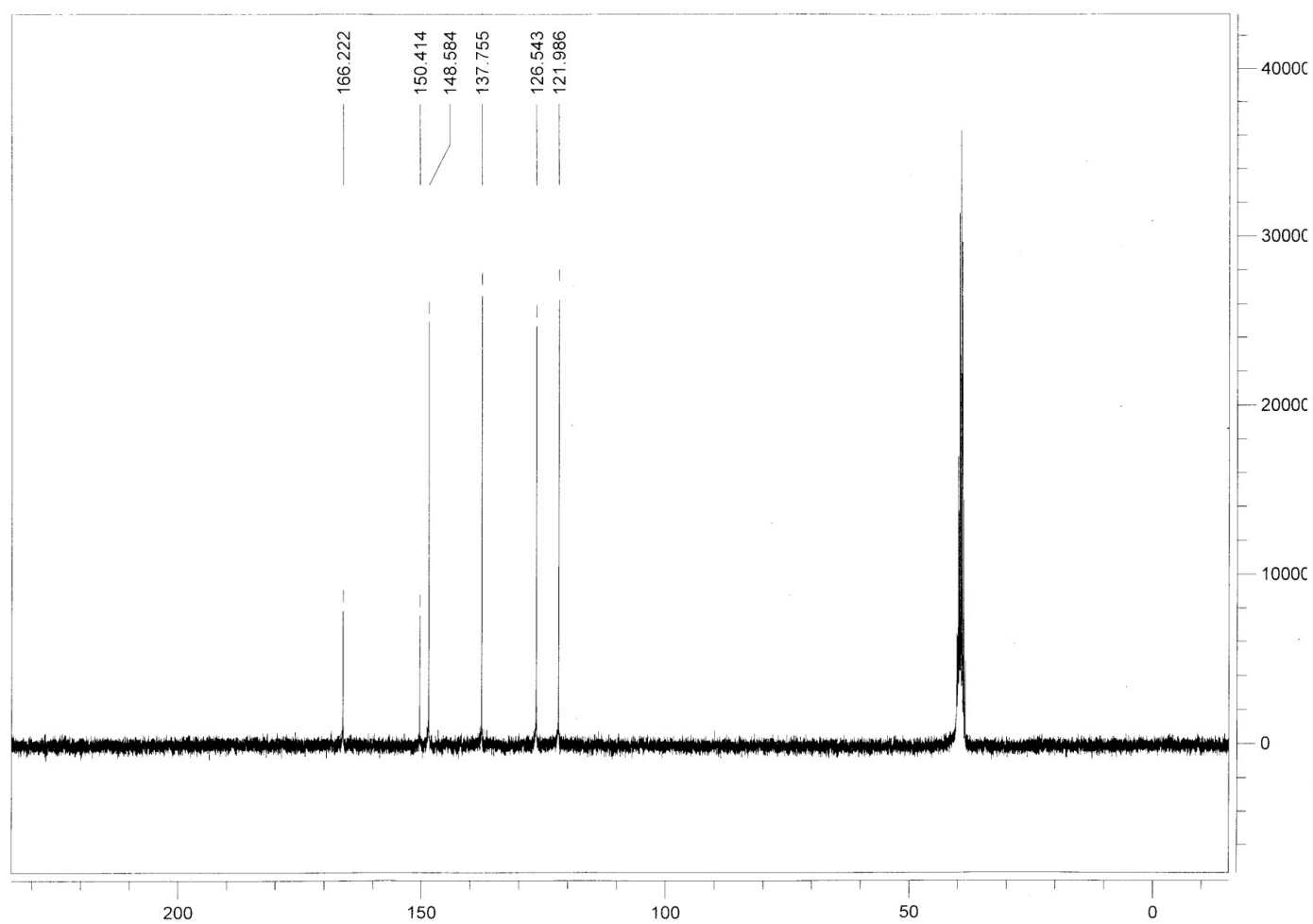


Figure S8. ^{13}C NMR spectrum of pic.

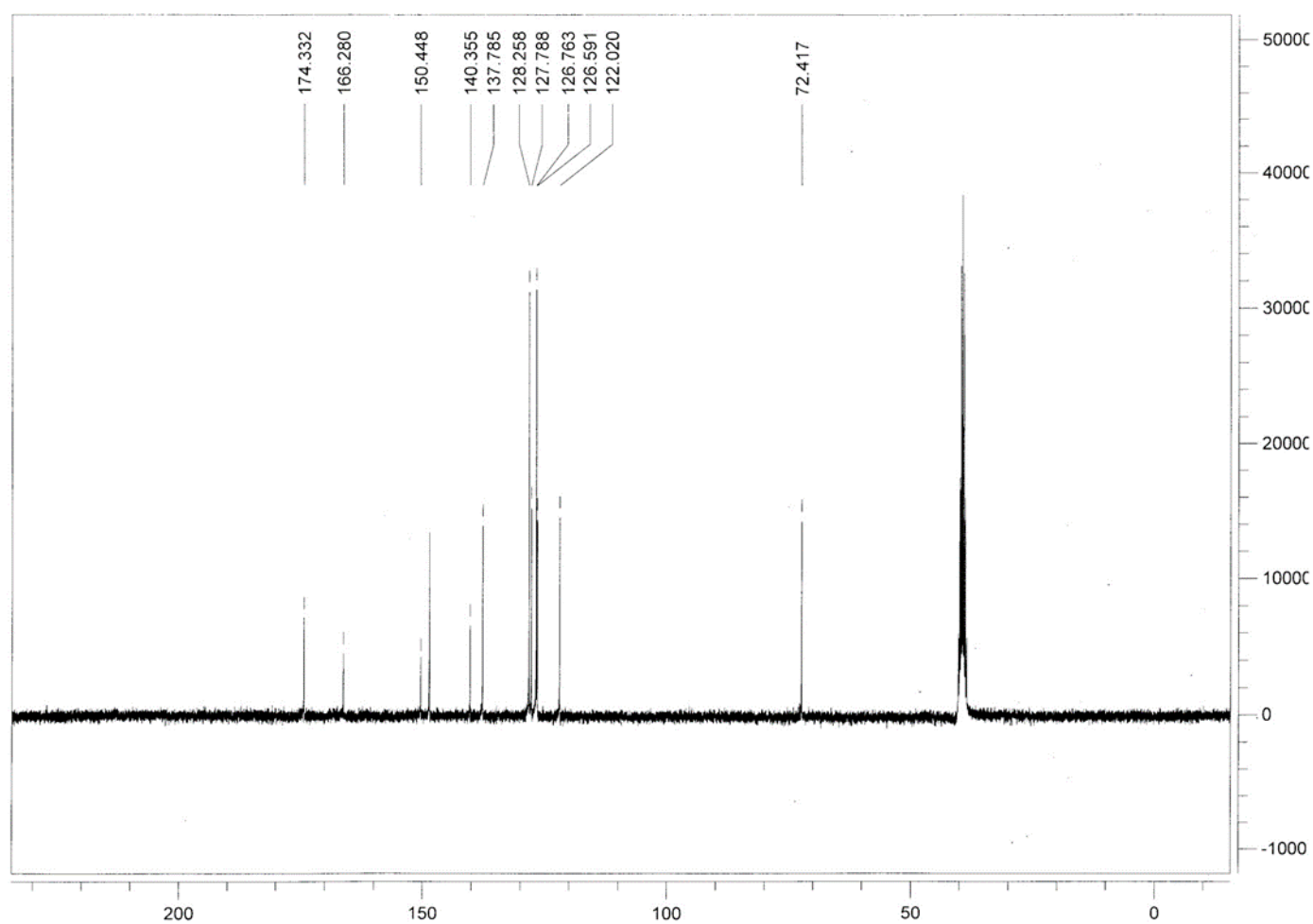


Figure S9. ^{13}C NMR spectrum of (pic)-(D-H₂ma) (1).

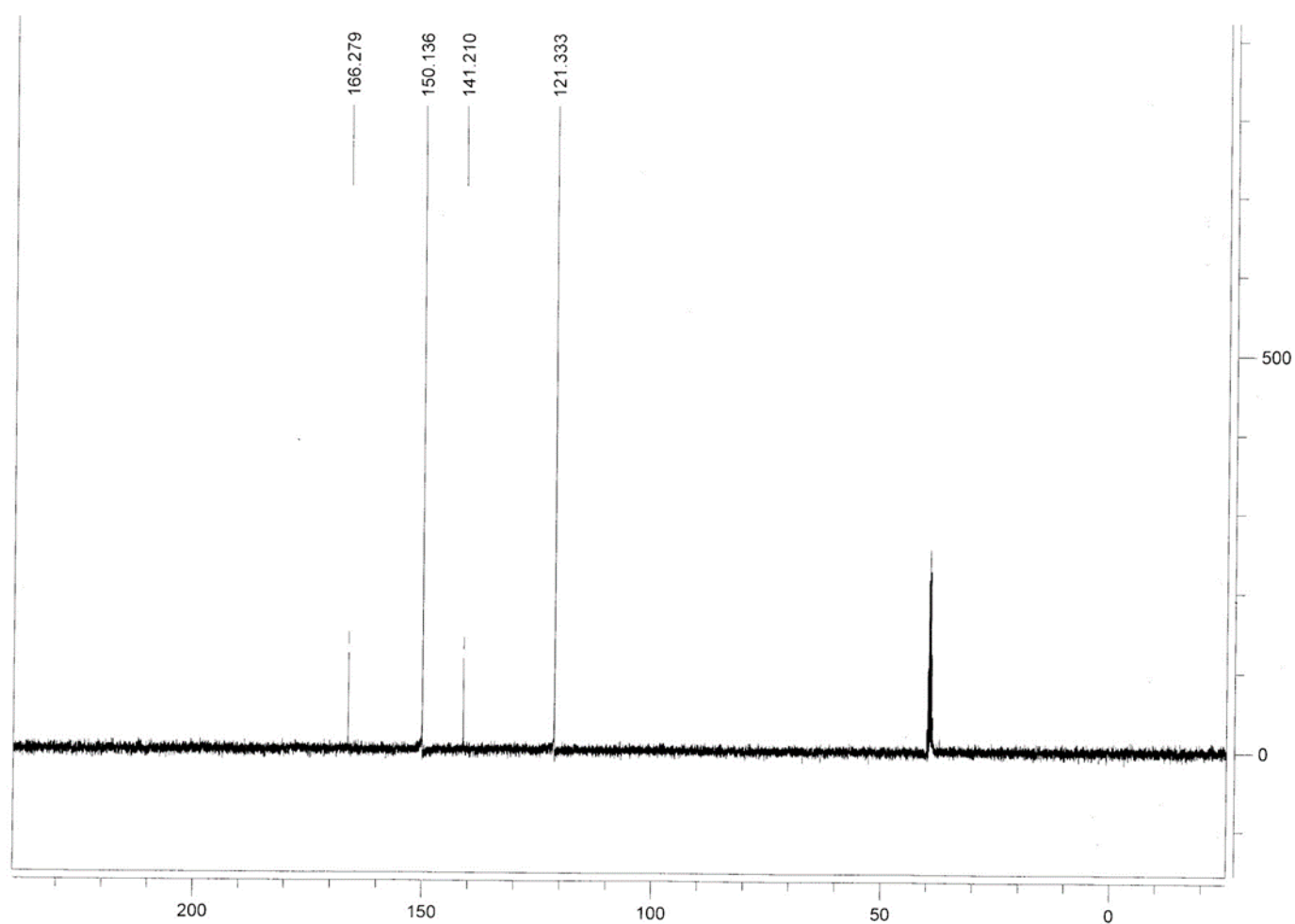


Figure S10. ^{13}C NMR spectrum of **inam**.

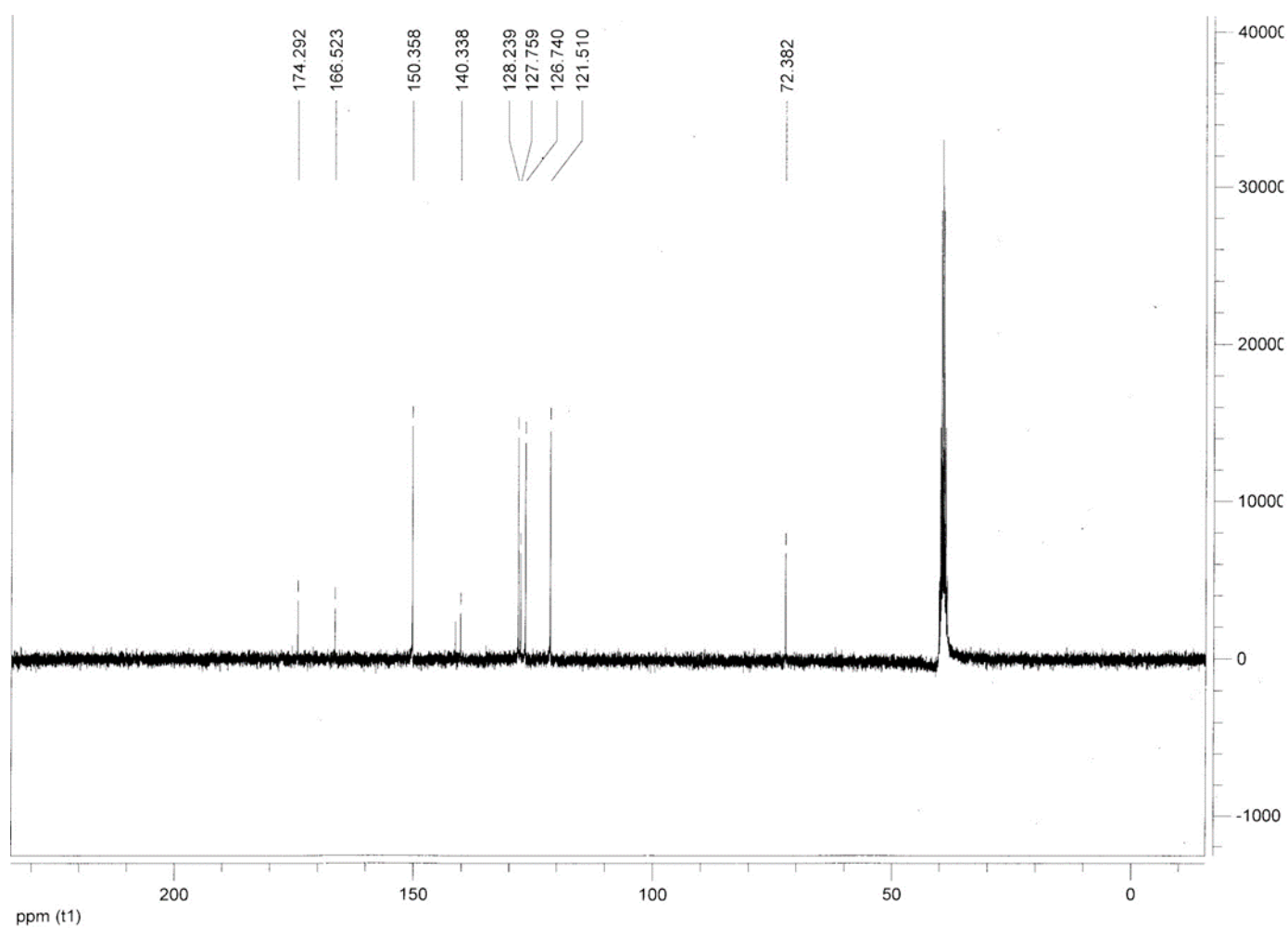


Figure S11. ^{13}C NMR spectrum of (inam)-(L-H₂ma) (3).

Table S1. Hydrogen bond parameters [\AA , $^\circ$] for cocrystals. Letters included as superscripts refer to symmetry codes shown in text and figures.

| Compound | D–H...A | D–H | H...A | D...A | DHA | Symmetry code |
|----------------------------------|-----------------------------|-----------|-----------|------------|-----------|---------------------|
| (pic)-(D-H ₂ ma) (1) | O11 H11...O10 ^a | 0.943(19) | 1.617(19) | 2.5472(13) | 168.3(17) | -x+1, -y, -z+1 |
| | O13 H13...N13 ^b | 0.846(19) | 2.374(19) | 3.0708(15) | 139.9(16) | x+1, y, z-1 |
| | N10 H10A...O12 ^a | 0.900(17) | 2.046(18) | 2.9287(16) | 166.6(15) | -x+1, -y, -z+1 |
| | N10 H10B...O12 ^c | 0.871(18) | 2.348(17) | 2.9479(15) | 126.3(14) | x-1, y, z+1 |
| HOGGOB [24] | O11 H11...O10 | 0.85(4) | 1.81(3) | 2.645(2) | 167(4) | |
| | O13 H13...N23 ^a | 0.85(4) | 2.36(4) | 3.005(3) | 133(3) | x+1, y+1, z |
| | N10 H10A...O12 | 0.86(3) | 2.10(3) | 2.946(3) | 168(3) | |
| | N10 H10B...O32 ^c | 0.91(4) | 2.22(4) | 2.951(3) | 137(3) | x, y+1, z |
| | O31 H31...O20 ^b | 0.83(4) | 1.74(4) | 2.554(2) | 166(4) | x+1, y, z |
| | O33 H33...O10 | 0.85(3) | 2.21(4) | 3.001(3) | 155(4) | |
| | N20 H20A...O32 ^d | 0.88(3) | 1.97(3) | 2.834(3) | 166(3) | x-1, y, z |
| | N20 H20B...O12 ^e | 0.86(4) | 2.20(4) | 2.927(3) | 142(3) | x-1, y-1, z |
| JILZOU01 (2a) [25] | O11 H11...N14 ^a | 0.852(19) | 1.835(19) | 2.6843(13) | 175.0(15) | x, y-1, z |
| | O13 H13A...O10 ^b | 0.834(18) | 1.993(16) | 2.7084(11) | 143.3(15) | -x,y-1/2,-z+1 |
| | N10 H10A...O12 ^c | 0.875(15) | 2.184(15) | 3.0020(12) | 155.4(15) | -x,y+1/2,-z+1 |
| | N10 H10B...O13 | 0.898(17) | 2.140(16) | 2.9212(13) | 144.9(13) | |
| | C13 H13...O10 ^e | 0.95 | 2.342 | 3.103 | 136.7 | x,y+1,z |
| JILZOU (2b) [26] | O11 H11...N24 ^a | 0.85(3) | 1.77(3) | 2.605(2) | 171(3) | -x+1/2, y+3/2, -z |
| | O13 H13A...O12 ^b | 0.84(3) | 1.93(3) | 2.6826(18) | 148(2) | -x+1/2, y-1/2, -z |
| | N10 H10A...O20 ^d | 0.88(3) | 2.06(3) | 2.902(2) | 159(2) | -x+1/2, y-1/2, -z+1 |
| | N10 H10B...O13 ^d | 0.88(2) | 2.21(2) | 3.0138(17) | 152(2) | |
| | O31 H31...N14 | 0.84(2) | 1.77(2) | 2.6046(18) | 176(2) | |
| | O33 H33...O31 ^c | 0.84(3) | 2.40(3) | 3.148(2) | 149(3) | x, y-1, z |
| | N20 H20A...O10 ^e | 0.88(3) | 2.06(3) | 2.932(2) | 169(2) | -x+1/2, y+1/2, -z+1 |
| | N20 H20B...O10 | 0.88(2) | 2.12(2) | 2.9734(18) | 162.0(19) | |
| (inam)-(L-H ₂ ma) (3) | O11 H11A...N15 ^a | 0.966(17) | 1.659(17) | 2.6241(14) | 177.3(16) | x-1, y, z |
| | O13 H13A...O10 ^b | 0.818(17) | 1.983(17) | 2.7750(14) | 162.9(16) | x, y, z-1 |
| | N10 H10A...O13 ^c | 0.851(15) | 2.234(15) | 2.9493(14) | 141.7(14) | -x+1, -y, -z+1 |
| | N10 H10B...O10 ^d | 0.913(15) | 1.969(16) | 2.8814(14) | 179.7(16) | -x, -y, -z+2 |
| | C16 H16...O12 ^e | 0.95 | 2.640 | 3.310 | 127.9 | x+1, y, z |
| | C17 H17...O12 ^c | 0.95 | 2.580 | 3.176 | 121.2 | -x+1, -y, -z+1 |

Crystallographic

The two crystals were obtained from the crystallization of solutions prepared re- 33 acting the pyridinecarboxamide isomers with DL-mandelic acid in a molar ratio 1:1. 34 Although the X-ray diffraction data were taken at 100 K, solid handling was always done at room temperature.

Diffraction data were obtained at 100(1) K using a Bruker X8 Kappa APEXII diffractometer from crystals mounted on glass fibers. Data were corrected for Lorentz and polarization effects and for absorption following multiscan [1] type. The structures were solved by direct methods [2], which revealed the positions of all nonhydrogen atoms. 40 These were refined on F^2 by a full-matrix least-squares procedure using anisotropic dis- 41 placement parameters [2]. Hydrogen atoms

were located in the difference maps and the positions of O–H and N–H hydrogen atoms were refined (others were included as riders); the isotropic displacement parameters of H atoms were constrained to 1.2 U_{eq} of the carrier atoms. Molecular graphics were generated with ORTEP [3] and DIAMOND [4].

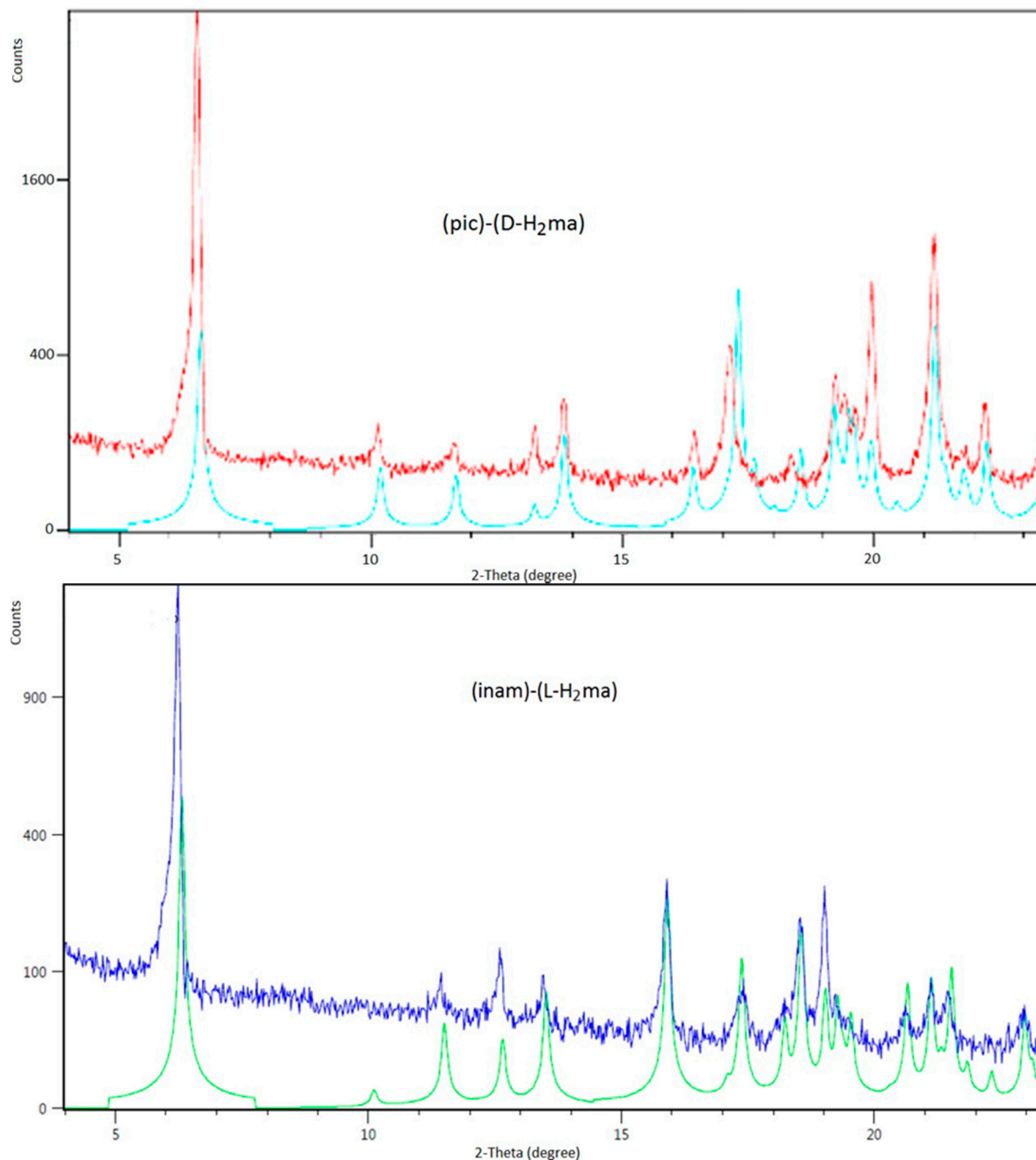


Figure S12. XRPD patterns of the solid forms of 1 and 3, obtained at room temperature. The XRPD-patterns of the cocrystals match well with the simulated XRPD. 49

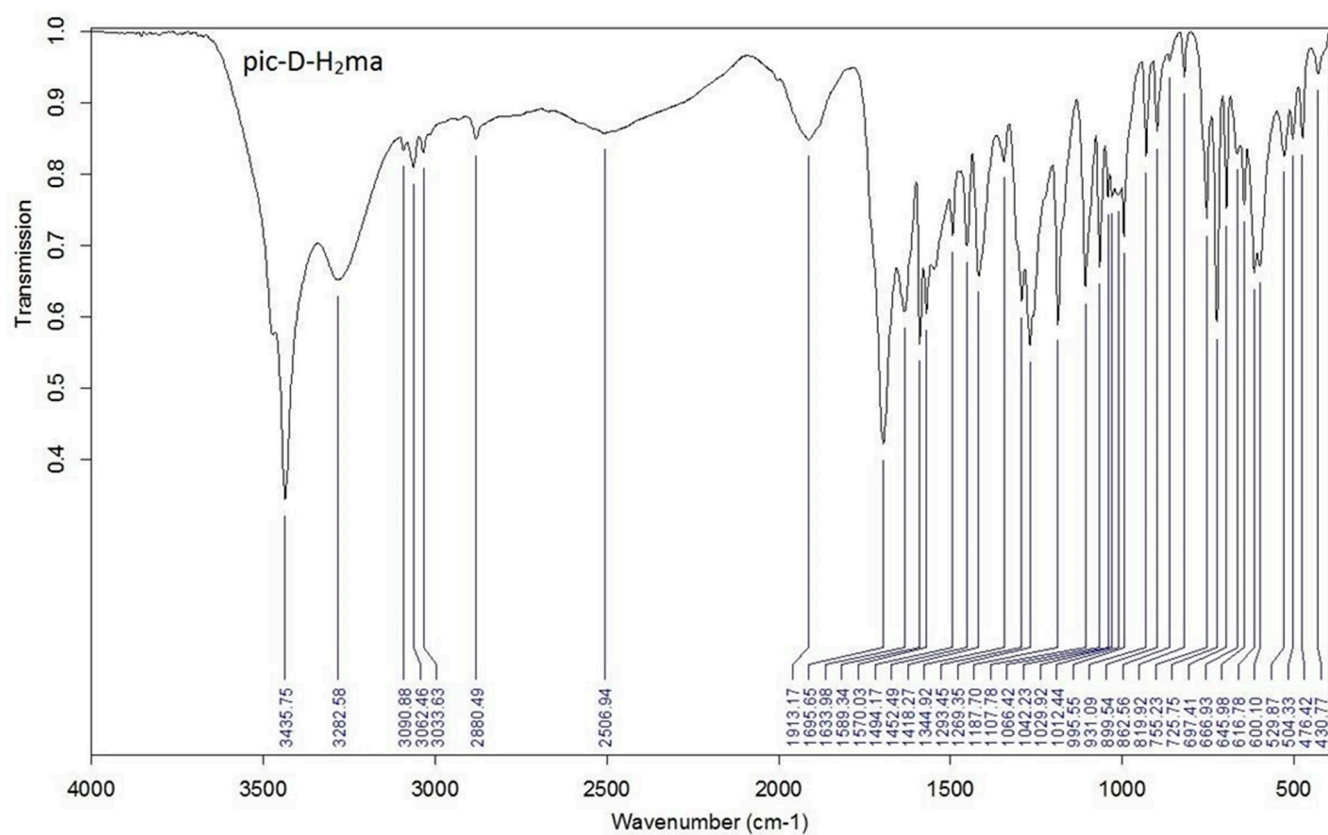


Figure S13. IR spectrum of (pic)-(D-H₂ma) (1).

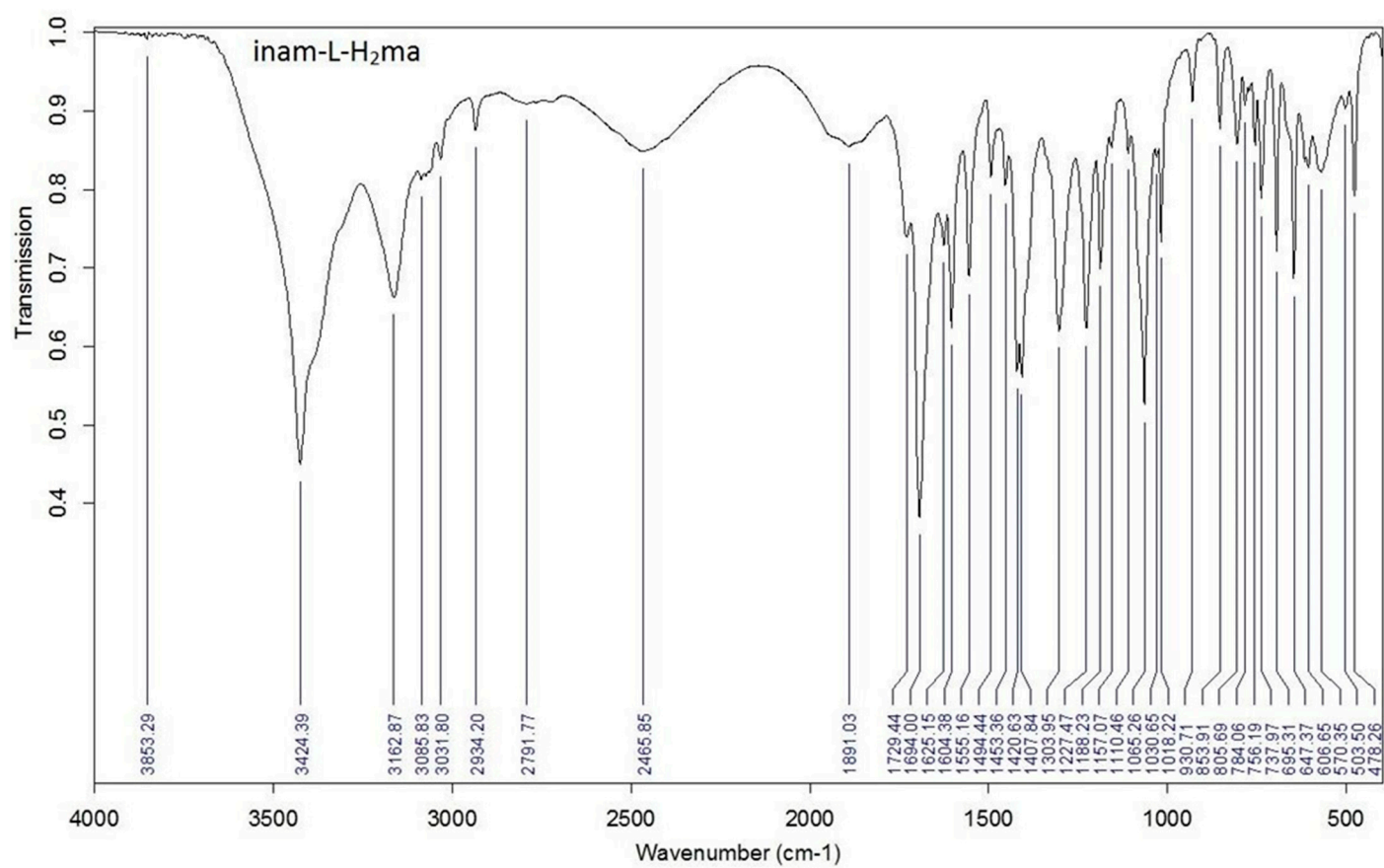


Figure S14. IR Spectrum of (inam)-(L-H₂ma) (3).