Synthesis of *anti* and *syn* Hydroxy-*iso*-Evoninic Acids

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Supporting Information — X-Ray Crystallography

The X-ray crystal structure of (–)-(2*R*,3*S*)-6a

Crystal data for (–)-(2*R*,3*S*)-**6a**: $C_{12}H_{17}NO_3$, M = 223.27, orthorhombic, $P2_12_12_1$ (no. 19), a = 7.5034(2), b = 12.2919(4), c = 13.0178(4) Å, V = 1200.65(6) Å^3, Z = 4, D_c = 1.235 g cm^{-3}, μ(Cu-Kα) = 0.725 mm^{-1}, T = 100 K, colourless laths, Agilent Technologies SuperNova diffractometer; 2448 independent measured reflections (R_{int} = 0.0413), F^2 refinement, R_1(obs) = 0.0302, wR_2(all) = 0.0760, 2287 independent observed absorption-corrected reflections [$|F_o| > 4\sigma(|F_o|)$, $2\theta_{max}$ = 149°], 153 parameters. The absolute structure of (-)-(2*R*,3*S*)-**2a** was determined by a combination of *R*-factor tests [R_1^+ = 0.0302, R_1^- = 0.0305], use of the Flack parameter [x^+ = 0.00(17), x^- = 1.02(17)] and determination using Bayesian statistics on Bijvoet differences (Hooft *et al.*, 2008), as implemented in the program PLATON (Spek, 2003). This gave probability values *p*3(ok), *p*3(twin) and *p*3(wrong) of 1.0, 0.4 x 10^{-6} and 0.6 x 10^{-25} respectively (1.0, 0.0, 0.0). The calculation was based on 5999 Bijvoet pairs. CCDC 869566.

The O(1) hydrogen atom was located from a Δ*F* map and refined freely.

The X-ray crystal structure of (+)-(2*S*,3*S*)-6b

Crystal data for (+)-(2*S*,3*S*)-**6b**: $C_{12}H_{17}NO_3$, M = 223.27, orthorhombic, $P2_12_12_1$ (no. 19), a = 5.97836(4), b = 12.67070(7), c = 15.76352(9) Å, V = 1194.087(12) Å^3, Z = 4, D_c = 1.242 g cm^{-3}, μ(Cu-Kα) = 0.729 mm^{-1}, T = 173 K, colourless shards, Oxford Diffraction Xcalibur PX Ultra diffractometer; 2366 independent measured reflections (R_{int} = 0.0283), F^2 refinement, R_1(obs) = 0.0262, wR_2(all) = 0.0726, 2337 independent observed absorption-corrected reflections [$|F_o| > 4\sigma(|F_o|)$, $2\theta_{max}$ = 145°], 152 parameters. The absolute structure of

(+)-(2*S*,3*S*)-**6b** was determined by a combination of *R*-factor tests [R_1^+ = 0.0262, R_1^- = 0.0264] and by use of the Flack parameter [x^+ = 0.00(16), x^- = 1.05(16)]. CCDC 869567.

The O(14) hydrogen atom was located from a Δ*F* map and refined freely subject to an O–H distance constraint of 0.90 Å.

Figures

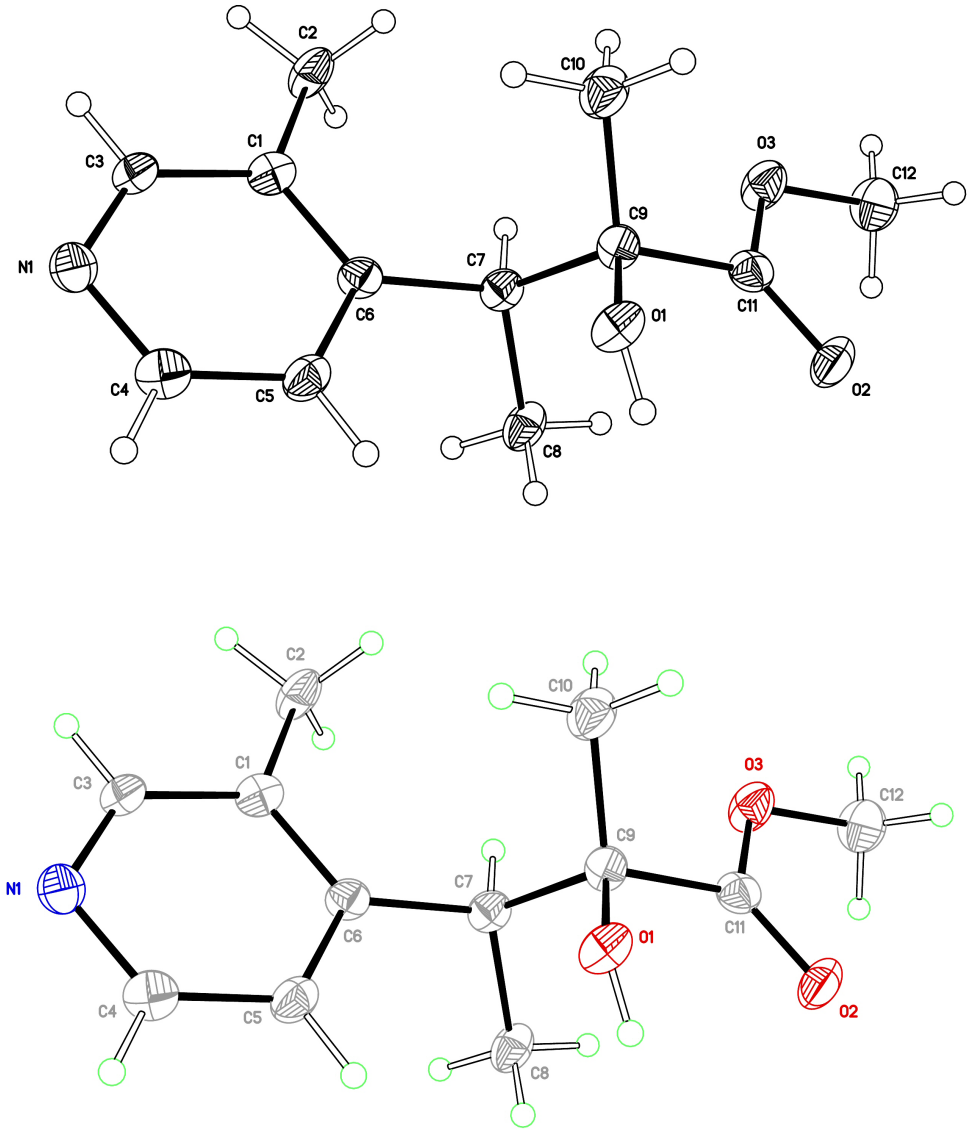

Figure S1. The molecular structure of (-)-**6a** showing the numbering scheme employed. Anisotropic atomic displacement ellipsoids for the non-hydrogen atoms are shown at the 50% probability level and hydrogen atoms are displayed as spheres of arbitrary radius.

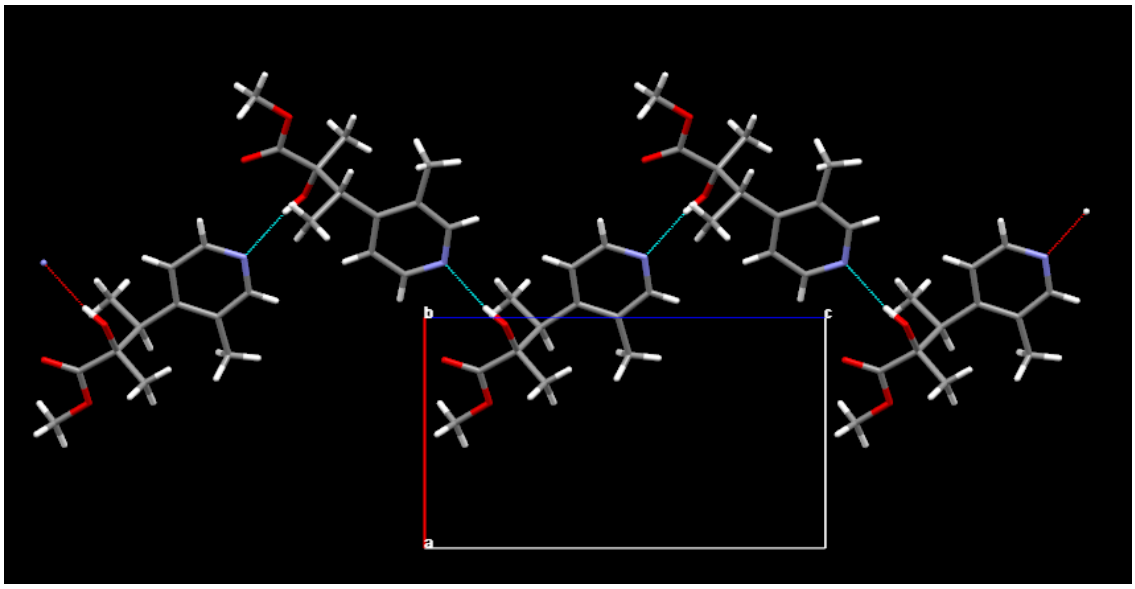

Figure S2. Single hydrogen bonded chain. View down the *b*-axis of (-)-**6a**.

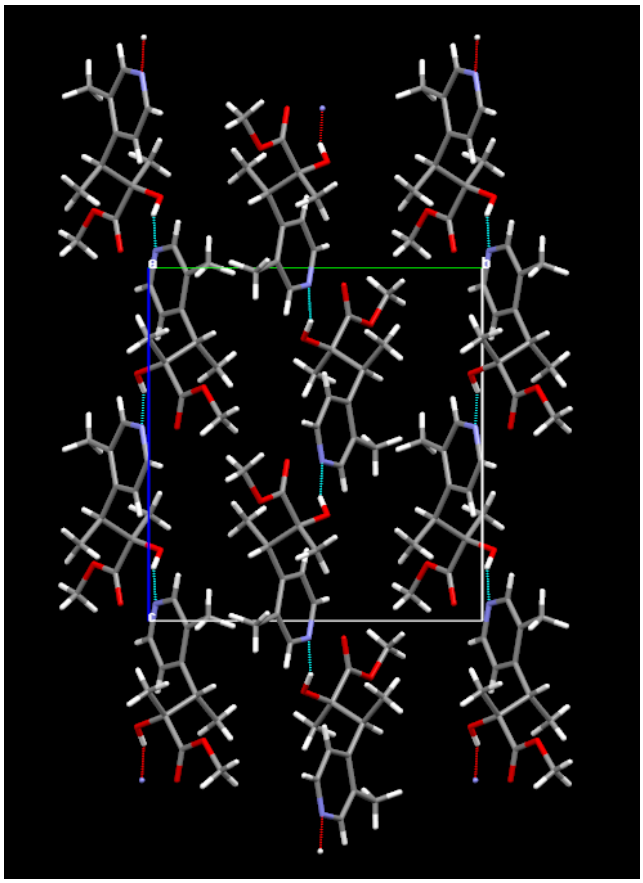

Figure S3. Crystal packing diagram. View down the *c*-axis of (-)-**6a**.

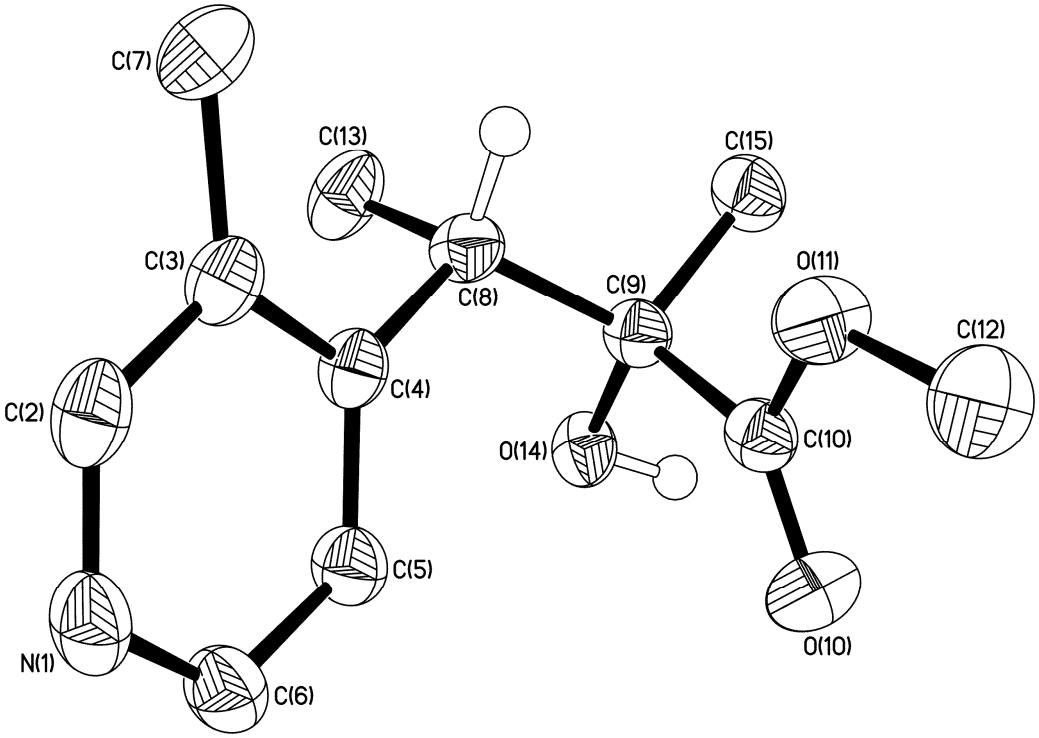

Figure S4. The crystal structure of (+)-(2*S*,3*S*)-**6b** (50% probability ellipsoids).

References

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