(±)-*trans, cis*-4-Hydroxy-5,6-di-*O*-isopropylidenecyclohex-2-ene-1-one: Synthesis and Facile Dimerization to Decahydrodibenzofurans

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Supporting Information — X-Ray Crystallography

Crystal data for **7**: $C_9H_{14}Br_2O_2$, M = 314.02, triclinic, P–1 (no. 2), a = 7.0842(10), b = 9.0032(13), c = 9.5389(6) Å, α = 95.640(8), β = 97.798(9), γ = 110.869(13)°, V = 556.12(13) Å^3, Z = 2, D_c = 1.875 g cm^{-3}, μ(Mo-Kα) = 7.256 mm^{-1}, T = 296 K, colourless blocks, Oxford Diffraction Xcalibur 3 diffractometer; 3660 independent measured reflections (R_{int} = 0.0261), F^2 refinement, R_1(obs) = 0.0315, wR_2(all) = 0.0635, 1964 independent observed absorption-corrected reflections [$|F_o| > 4\sigma(|F_o|)$, $2\theta_{max}$ = 66°], 119 parameters. CCDC 799140.

Crystal data for **8**: $C_{18}H_{24}O_8$, M = 368.37, orthorhombic, $Pca2_1$ (no. 29), a = 13.3473(10), b = 14.7931(14), c = 17.9988(19) Å, V = 3553.8(6) Å^3, Z = 8 [two independent molecules], D_c = 1.377 g cm^{-3}, μ(Cu-Kα) = 0.915 mm^{-1}, T = 173 K, colourless prisms, Oxford Diffraction Xcalibur PX Ultra diffractometer; 6380 independent measured reflections (R_{int} = 0.0385), F^2 refinement, R_1(obs) = 0.0478, wR_2(all) = 0.1224, 5848 independent observed absorption-corrected reflections [$|F_o| > 4\sigma(|F_o|)$, $2\theta_{max}$ = 139°], 477 parameters. The absolute structure of **8** could not be unambiguously determined by either *R*-factor tests [R_1^+ = 0.0478, R_1^- = 0.0479] or by use of the Flack parameter [x^+ = +0.28(13), x^- = +0.72(13)]. CCDC 798850.

Compound **8** crystallised with two independent molecules (A and B) in the asymmetric unit. The two molecules have essentially identical conformations with an r.m.s. fit of all the non-hydrogen atoms of ca. 0.06 Å (see Fig. S6 in the supporting information). The hydroxy proton in each molecule was located from a ΔF map and refined freely subject to an O–H distance constraint of 0.90 Å.

Figures

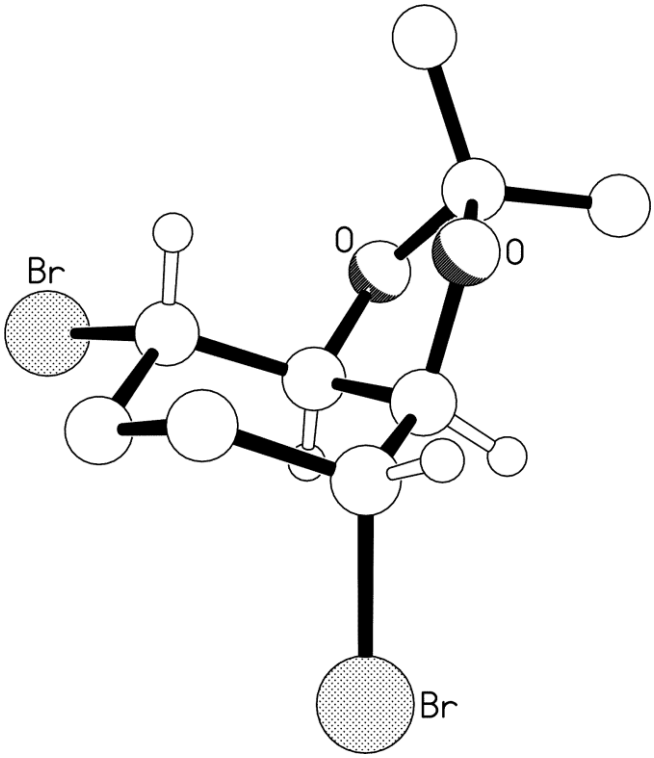

Fig. S1 The molecular structure of **7**.

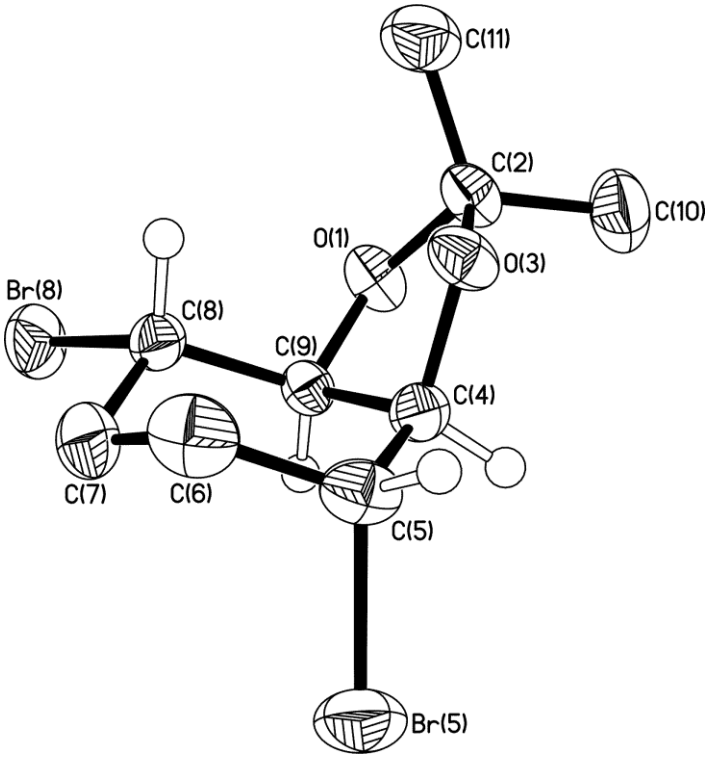

Fig. S2 The molecular structure of **7** (50% probability ellipsoids).

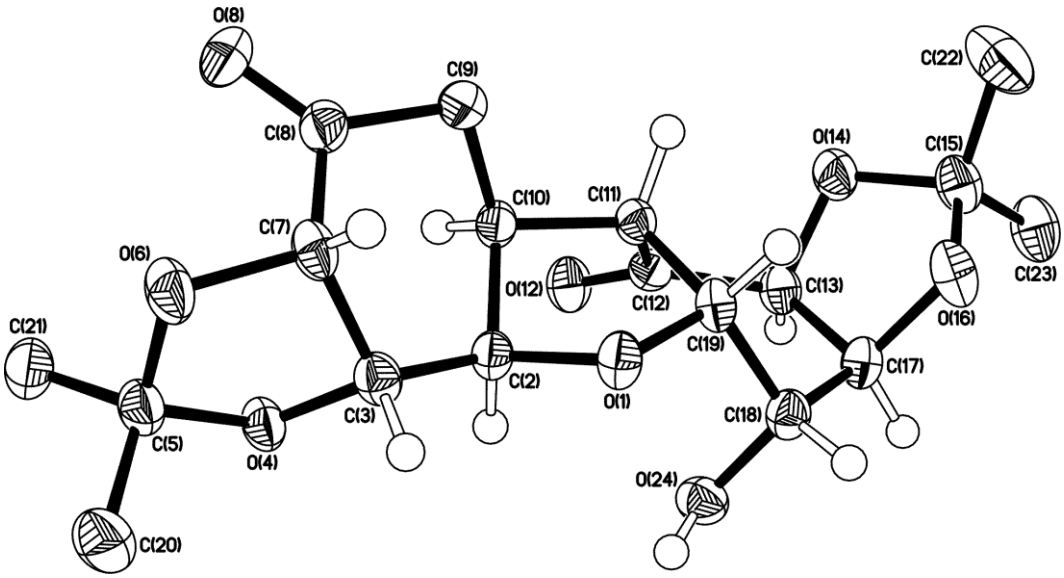

Fig. S3 The molecular structure of one (**8**-**A**) of the two crystallographically independent molecules present in the crystals of **8** (50% probability ellipsoids).

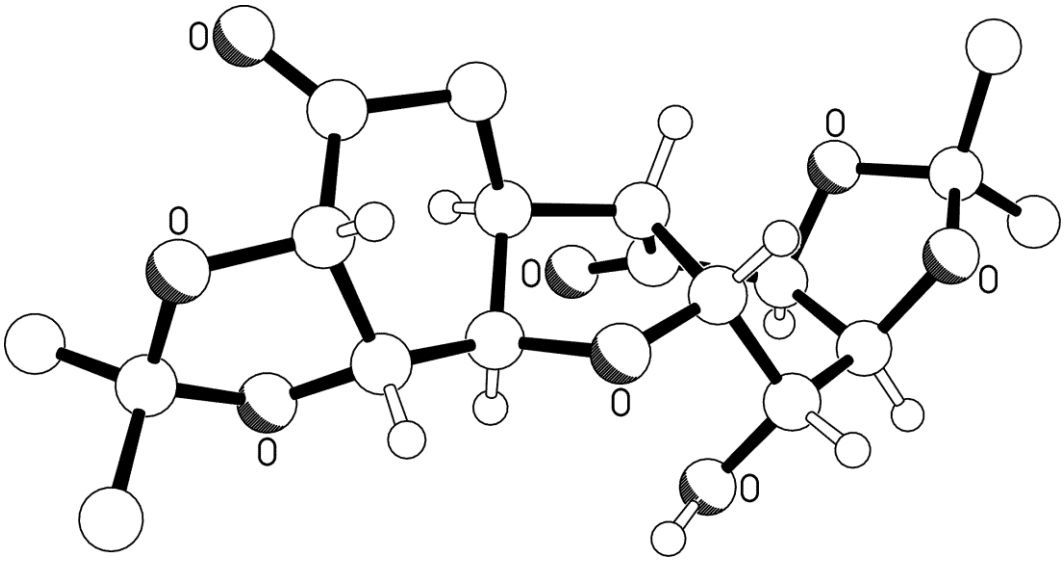

Fig. S4 The molecular structure of one (**8**-**B**) of the two crystallographically independent molecules present in the crystals of **8**.

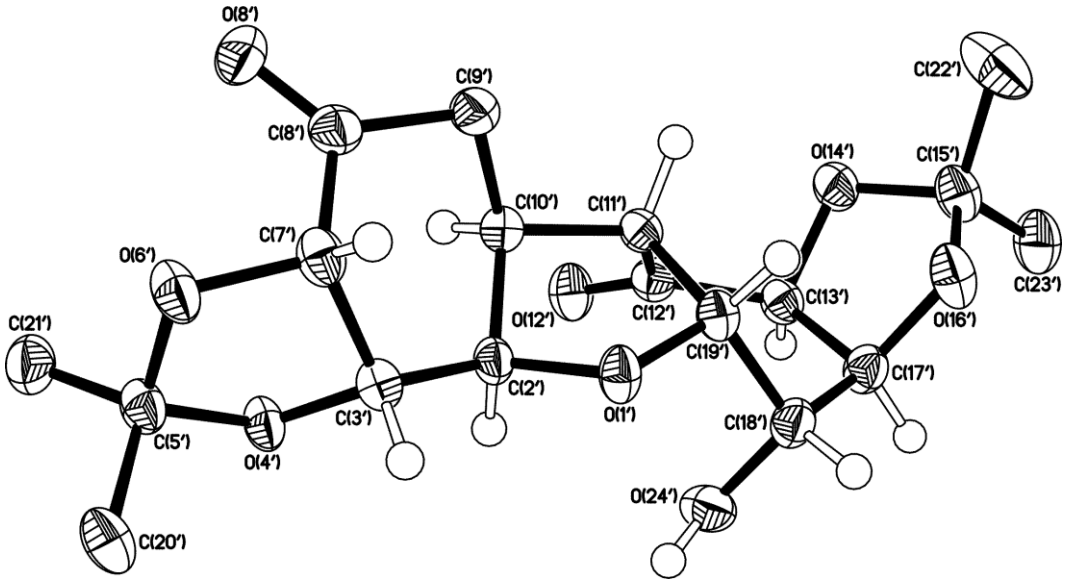

Fig. S5 The molecular structure of one (**8**-**B**) of the two crystallographically independent molecules present in the crystals of **8** (50% probability ellipsoids).

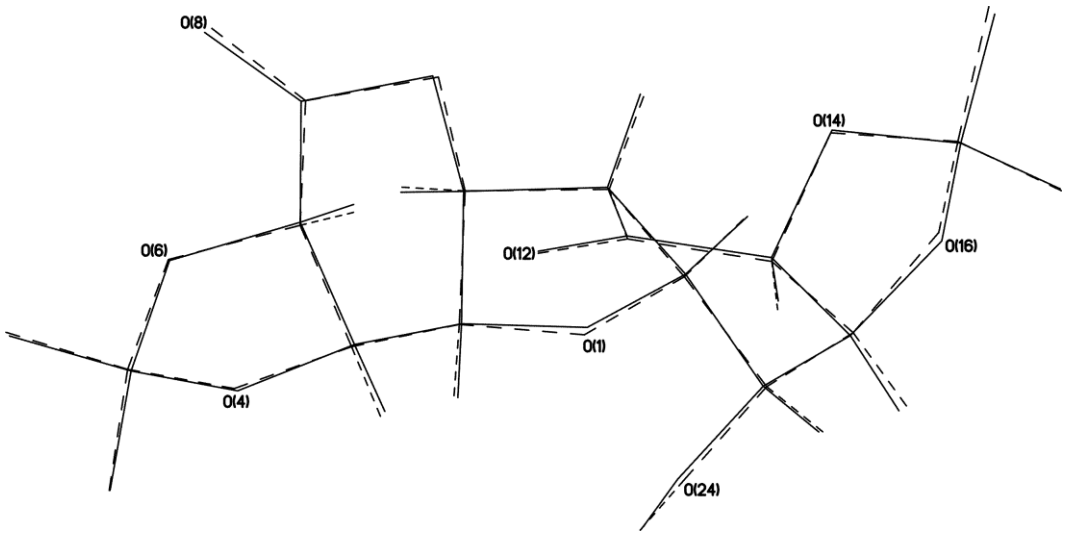

Fig. S6 Overlay of the two crystallographically independent molecules present in the crystals of **8** (molecule **A** has been drawn with line bonds, and molecule **B** with dashed bonds).