Supplementary Material (ESI) for Chemical Communications

Temperature-dependent crystal structure of the isopropanol clathrate of Dianin's compound

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Electronic Supplementary Information

Experimental:

Dianin's compound was prepared from a literature procedure (W. Baker, A. J. Floyd, J. F. W. McOmie, G. Pope, A. S. Weaving and J. H. Wild, *J. Chem. Soc.*, 1956, 2010-2017). Clathrate complexes were prepared by recrystallisation of free Dianin's compound (50 mg, 0.19 mol) from the desired guest which is a liquid at room temperature. Minimal amount of solvent was added, the solution filtered, then left to crystallise by slow evaporation.

Laboratory X-ray data were measured from single crystals using Oxford Diffraction Xcalibur or Gemini CCD diffractometers with monochromatic MoKα radiation ($\lambda = 0.71073$Å). The structures were solved by direct methods and refined on full matrix least-squares procedures on F^2. A full matrix least-squares refinement procedure was used, minimising $w(F_o^2 - F_c^2)$, with $w = [\sigma^2(F_o^2) + (AP)^2 + BP]^{-1}$, where $P = (F_o^2 + 2F_c^2)/3$. Agreement factors ($R1 = \sum \| |F_o| - |F_c| \| / \sum |F_o|$, $wR2 = \left\{ \sum \left[w(F_o^2 - F_c^2)^2 \right] / \sum \left[w(F_o^2)^2 \right] \right\}^{1/2}$ and $\mathrm{GOF} = \left\{ \sum \left[w(F_o^2 - F_c^2)^2 \right] / (n-p) \right\}^{1/2}$ are cited where n is the number of reflections and p is the total number of parameters refined. Non-hydrogen non-disordered atoms were refined anisotropically. Some restraints were applied during refinement of the guest molecules for the reason of keeping their chemical integrity and to avoid the symmetry breaking. The positions of hydrogen atoms were partly localised from different Fourier synthesis, partly calculated from geometrical consideration and their atomic parameters were constrained to the bonded atoms during the refinement with O–H = 0.85 Å , C_{ph}–H = 0.95 Å, C_{Me}–H = 0.98 Å, and 0.99 Å in CH_2.

Crystal data for guest-free DC:

$C_{18}H_{20}O_2$, M = 268.34, colorless needle, 0.24 × 0.19 × 0.14 mm^3, trigonal, space group $R\bar{3}$ (No. 148), a = b = 26.7783(12) Å, c = 10.9031(4) Å, V = 6770.9(4) Å^3, Z = 18, D_c = 1.185 g/cm^3, μ = 0.076 mm^{-1}. F_{000} = 2592, MoKα radiation, λ = 0.71073 Å, T = 100(2) K, $2\theta_{max}$ = 64.6º, 48523 reflections collected, 5194 unique (R_{int} = 0.0449). Final GOF = 1.003, $R1$ = 0.0479, $wR2$ = 0.1224, R indices based on 3511 reflections with $I > 2\sigma(I)$ (refinement on F^2), 181 parameters, 0 restraints. Lp and absorption corrections applied. CCDC 796805

Crystal data for DC:isopropanol at four temperatures:

***T* = 299 K**: $C_{57}H_{68}O_7$, M = 865.11, colorless prism, 0.42 × 0.33 × 0.21 mm^3, trigonal, space group $R\bar{3}$ (No. 148), a = b = 27.2014(10) Å, c = 11.0239(3) Å, V = 7064.0(3) Å^3, Z = 6, D_c = 1.220 g/cm^3, μ = 0.079 mm^{-1}. F_{000} = 2796, MoKα radiation, λ = 0.71073 Å, T = 299(2) K, $2\theta_{max}$ = 64.5º, 26572 reflections collected, 5279 unique (R_{int} = 0.0205). Final GOF = 1.002, $R1$ = 0.0609, $wR2$ = 0.1786, R indices based on 3998 reflections with $I > 2\sigma(I)$ (refinement on F^2), 198 parameters, 2 restraints. Lp and absorption corrections applied. CCDC 796806

***T* = 182 K**: $C_{57}H_{68}O_7$, M = 865.11, colorless prism, 0.42 × 0.33 × 0.21 mm^3, trigonal, space group $R\bar{3}$ (No. 148), a = b = 27.0106(4) Å, c = 10.9760(1) Å, V = 6934.94(12) Å^3, Z = 6, D_c = 1.243 g/cm^3, μ = 0.080 mm^{-1}. F_{000} = 2796, MoKα radiation, λ = 0.71073 Å, T = 182(2)K, $2\theta_{max}$ = 64.5º, 26137 reflections collected, 5148 unique (R_{int} = 0.0153). Final GOF = 1.003, $R1$ = 0.0617, $wR2$ = 0.1729, R indices based on 4279 reflections with $I > 2\sigma(I)$ (refinement on F^2), 197 parameters, 2 restraints. Lp and absorption corrections applied. CCDC 796807

***T* = 100 K**: $C_{57}H_{68}O_7$, M = 865.11, colorless prism, 0.42 × 0.33 × 0.21 mm^3, trigonal, space group $R\bar{3}$ (No. 148), a = b = 53.9718(10) Å, c = 11.0324(3) Å, V = 27831.3(9) Å^3, Z = 24, D_c = 1.239 g/cm^3, μ = 0.080 mm^{-1}. F_{000} = 11184, MoKα radiation, λ = 0.71073 Å, T = 100(2)K, $2\theta_{max}$ = 56.6º, 64482 reflections collected, 14214 unique (R_{int} = 0.0214). Final GOF = 1.005, $R1$ = 0.0501, $wR2$ = 0.1221, R indices based on 11522 reflections with $I > 2\sigma(I)$ (refinement on F^2), 793 parameters, 1 restraint. CCDC 796808

***T* = 15 K**: $C_{57}H_{68}O_7$, M = 865.11, colorless needle, 0.08 × 0.03 × 0.02 mm^3, trigonal, space group $R\bar{3}$ (No. 148), a = b = 53.7098(13) Å, c = 10.9911(3) Å, V = 27458.6(10) Å^3, Z = 24, D_c = 1.256 g/cm^3, μ = 0.052 mm^{-1}. F_{000} = 11184, Bruker APEX II, synchrotron radiation at Argonne Advanced Photon Source (APS), λ = 0.41328 Å, T = 15(2) K, $2\theta_{max}$ = 57.1º, 835170 reflections collected, 70274 unique (R_{int} = 0.0488). Final GOF = 1.057, $R1$ = 0.0669, $wR2$ = 0.1711, R indices based on 53909 reflections with $I > 2\sigma(I)$ (refinement on F^2), 830 parameters, 1 restraint. CCDC 796809