Trimeric liquid crystals assembled using both hydrogen and halogen bonding

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Electronic Supplementary Information

Crystals **2**-6 and **4**-1 had data collected on Bruker SMART[1] Apex X-ray diffractometer at 110 K, controlled by Bruker SMART and integrated using Bruker SAINT+ software,[2] whilst crystal **3** had data collected on a Bruker Nonius KappaCCD Area Detector at the window of a Bruker Nonius FR591 rotating anode driven by COLLECT[3] and DENZO[4] software at 120 K. All the structures were determined in SHELXS-97[5] and refined using SHELXL-97[6] with full-matrix least squares on F^2. All non-hydrogen atoms were refined anisotropically. For **2**-6 and **4**-1, the hydrogens were placed using a riding model except for the hydroxyl hydrogen which was placed by difference map after all other atoms were located. For **3** the hydrogen atoms were found by difference map and allowed to refine isotropically.

Crystal Data for 2-6

Crystal data for **2**-6: $C_{44}H_{47}F_4IN_2O_3$; M = 854.74 g mol^{-1}; T = 110(2) K; λ = 0.71073 Å; triclinic, space group P–1; a = 10.4105(10), b = 10.9417(11), c = 19.4274(19) Å; α = 75.531(2), β = 78.768(2), γ = 66.626(2); V = 1955.5(3) Å^3; Z = 2; ρ_{calc} = 1.452 Mg m^{-3}; μ(Mo-K$_\alpha$) = 0.880 mm^{-1}; F(000) = 876; crystal size = 0.38 x 0.10 x 0.06 mm. A total of 19211 reflections were collected (1.09 ≤ θ ≤ 28.31°) of which 9443 were independent (R_{int} = 0.0281). The structure was solved using SHELXS-97 and refined using SHELXL-97 with full-matrix least squares on F^2, 499 parameters, GoF = 1.117, R_1[I>2σ(I_0)] = 0.0442, wR_2[I>2σ(I_0)] = 0.1159; R_1 (all reflections) = 0.0579, wR_2 (all reflections) 0.1320; –1.119 < $\Delta\rho$ < 1.836 eÅ^3. CCDC xxxyyy. For crystallographic data in CIF and other electronic format see DOI: xxxxx.

Crystal Data for 3

Crystal data for **3**: $C_{31}H_{36}F_5NO_2$; M = 549.61 g mol^{-1}; T = 120(2) K; λ = 0.71073 Å; triclinic, space group P–1; a = 6.0419(4), b = 14.9401(11), c = 16.9981(13) Å; α = 109.712(4), β = 99.798(3), γ = 101.546(4)°; V= =1367.42(17) Å^3; Z = 2; ρ_{calc} = 1.335 Mg m^{-3}; μ(Mo-K$_\alpha$) = 0.105 mm^{-1}; *F(000)* = 580; crystal size = 0.23 × 0.10 × 0.08 mm^3. The crystals were stacked plates that gave a slightly smeared diffraction pattern, especially at the higher angles, but was perfectly acceptable to solve, resulting in a high R_{int} and R_1 (all data). A total of 22968 reflections were collected (2.63 ≤ θ ≤ 27.55°) of which 6154 were independent (R_{int} = 0.1539). The structure was solved using SHELXS-97 and refined using SHELXL-97 with full-matrix least squares on F^2, 496 prameters, GoF = 0.913; R_1[I>2σ(I_0)] = 0.0610, wR_2[I>2σ(I_0)] = 0.1034; R_1 (all reflections) = 0.2173, wR_2 (all reflections) 0.1406; –0.281 < $\Delta\rho$ < 0.258 eÅ^3. CCDC xxxyyy. For crystallographic data in CIF and other electronic format see DOI: xxxxx.

Crystal Data for 4-**1**

Crystal data for **4**-1: $C_{20}H_{14}F_4INO_2$; 503.22 g mol^{-1}; T = 110(2) K; λ = 0.71073 Å; orthorhombic, space group $P2_12_12_1$; a = 6.0363(5), b = 8.5988(6), c = 35.409(3) Å; α = β = γ = 90°; V= 1837.9(3) Å^3; Z = 4; ρ_{calc} = 1.819 Mg m^{-3}; μ(Mo-K$_\alpha$) = 1.798 mm^{-1}; *F(000)* = 984; crystal size = 0.27 × 0.23 × 0.04 mm^3. A total of 20820 reflections were collected ($2.30 \leq \theta \leq 30.01°$) of which 5331 were independent (R_{int} = 0.0358). The structure was solved using SHELXS-97 and refined using SHELXL-97 with full-matrix least squares on F^2, 277 parameters, *GoF* = 1.134; $R_1[I>2\sigma(I_0)]$ = 0.0377, w$R_2[I>2\sigma(I_0)]$ = 0.0841; R_1 (all reflections) = 0.0441, wR_2 (all reflections) 0.0865; –1.103 < $\Delta\rho$ < 1.046 eÅ^3. CCDC xxxyyy. For crystallographic data in CIF and other electronic format see DOI: xxxxx.

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1 SMART diffractometer control software (v5.625), Bruker-AXS GMBH, Karlsruhe, Germany.

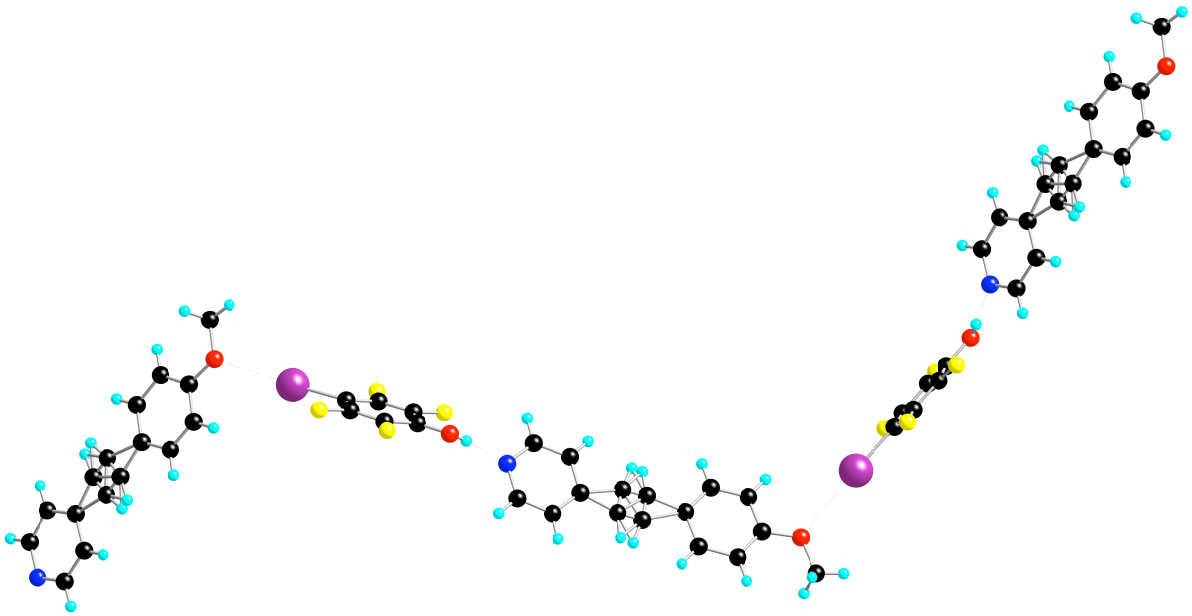
2 Saint+ integration and absorption-correction software (v6.22) , Bruker-AXS GMBH, Karlsruhe, Germany.

3 Collect: Data collection software, R. Hooft, Nonius B.V., 1998

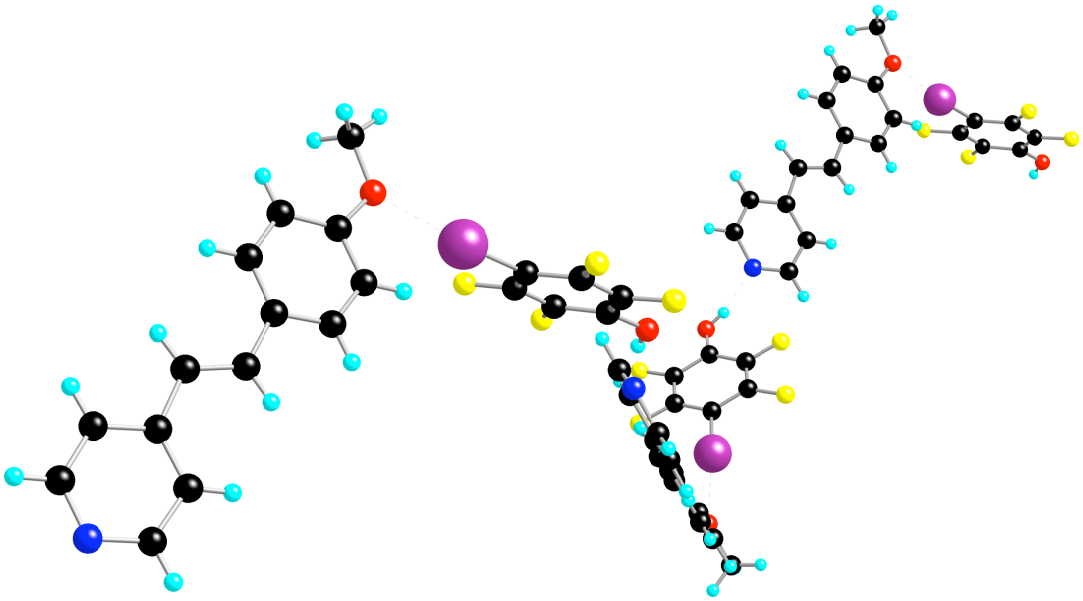
4 Z. Otwinowski & W. Minor, Methods in Enzymology (1997) Vol. 276: Macromolecular Crystallography, part A, pp. 307-326; C. W. Carter, Jr. & R. M. Sweet, Eds., Academic Press

5 G. M. Sheldrick, Acta Cryst. (1990) A46 467-473

6 G. M. Sheldrick (1997), University of Göttingen, Germany



Representation of the disorder in the structure of **4**-1



Helical arrangement in the structure of **4**-1.