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Supporting information for article:

Single crystal X-ray structure determination and temperature-dependent structural studies of the smectogenic compound 7OS5

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Supporting information

Single-crystal X-ray diffraction

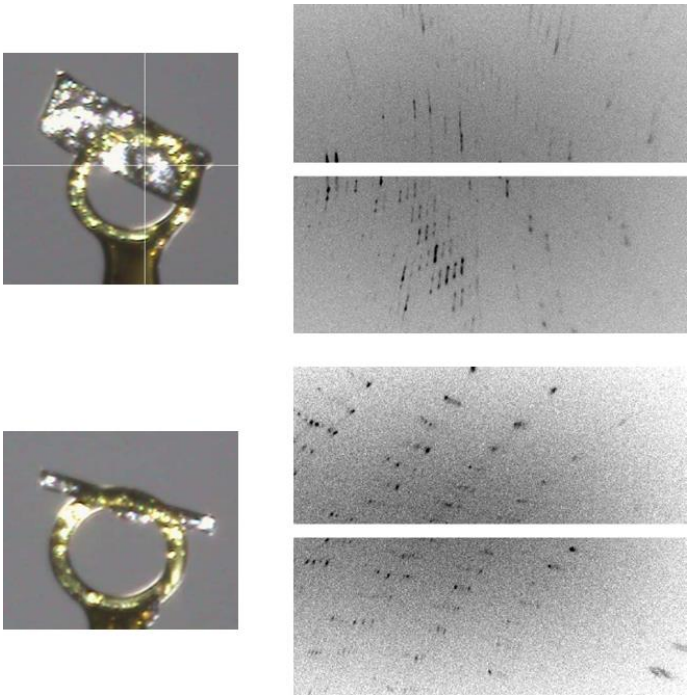


Figure S1 Two 7OS5 crystals (sized 0.50 mm × 0.08 mm × 0.02 mm and 0.36 mm × 0.03 mm × 0.01 mm – the top one and the bottom one, respectively) and their SC-XRD patterns proving that they are actually aggregates of a number of crystallites (XtaLAB P200 MM007-DW (Rigaku) single crystal diffractometer; CuKα; PILATUS 200k detector).

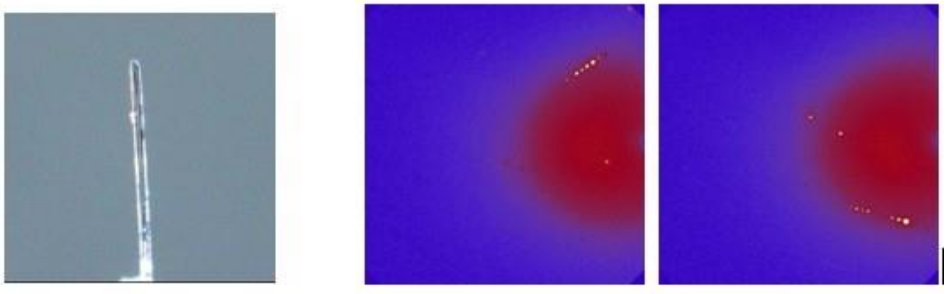


Figure S2 The small, needle-shaped (sized 0.38 mm × 0.02 mm × 0.01 mm) single crystal of 7OS5 selected for structure determination with its diffraction pattern (SuperNova (Agilent Technologies) diffractometer; CuKα; Atlas CCD detector (2x2k) and mirror monochromator). The long axis of the needle is along the *b* direction.

Polarizing optical microscopy (POM)

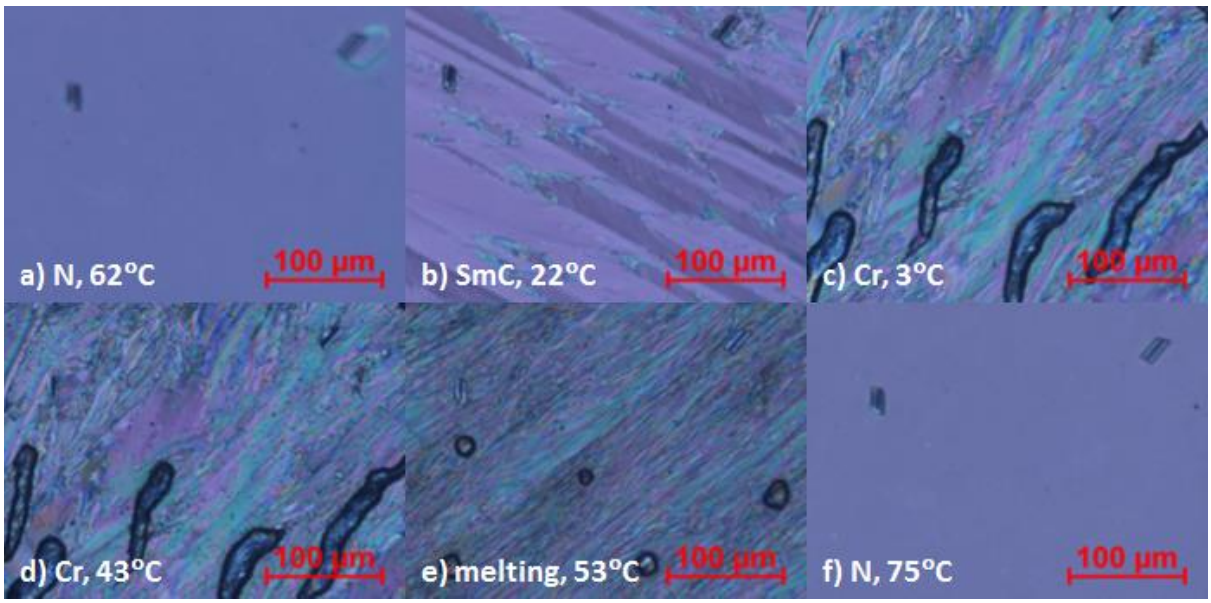


Figure S3 Textures of 7OS5 registered during cooling from isotropic liquid (a-c) and subsequent heating (d-f) at the 6°C min^{-1} rate.

Principal axis strain calculations

Principal axis strain calculator PASCal (http://pascal.chem.ox.ac.uk) was used for analysis of the temperature variation of the lattice parameters of 7OS5 single crystal on heating.

Table S1. Input data for PASCal.

phase	T [K]	ΔT [K]	*a* [Å]	*b* [Å]	*c* [Å]	α [deg]	β [deg]	γ [deg]
Cr I	233	0.5	54.31	5.6000	15.516	90	90	90
	253	0.5	54.36	5.6011	15.606	90	90	90
	273	0.5	54.25	5.6072	15.714	90	90	90
	293	0.5	54.20	5.6067	15.841	90	90	90
	308	0.5	54.10	5.6112	15.949	90	90	90
	320	0.5	54.00	5.6155	16.070	90	90	90
Cr II	113	0.5	54.25	5.5608	15.157	90	94.42	90
	133	0.5	54.29	5.5608	15.190	90	94.14	90
	153	0.5	54.35	5.5724	15.251	90	93.92	90
	173	0.5	54.33	5.5714	15.285	90	93.62	90
	193	0.5	54.28	5.573	15.333	90	93.24	90
	213	0.5	54.41	5.5852	15.439	90	92.31	90

Table S2. Coefficients of thermal expansion (CTEs) along principal directions x_i, volume principal thermal expansivity and the relationship between the principal axes and the crystallographic axes.

phase	i-th principal axis	α_i [$10^{-6}K^{-1}$]	x_i component along the crystallographic axes *a*	*b*	*c*
Cr I	1	-65(12)	-1.0000	0.0000	0.0000
	2	27(4)	0.0000	1.0000	0.0000
	3	404(24)	0.0000	0.0000	1.0000
	V	367(15)			
Cr II	1	-80(14)	-0.4216	0.0000	0.9068
	2	41(7)	0.0000	1.0000	0.0000
	3	297(37)	0.1782	0.0000	0.9840
	V	277(29)			

Cr I phase: area CTE: $\alpha_2 + \alpha_3 = 431(28)\cdot10^{-6}K^{-1}$ and linear CTE: $\alpha_1 = -65(12)\cdot10^{-6}K^{-1}$

the principal axes coincide with the crystallographic axes

Cr II phase: area CTE: $\alpha_2 + \alpha_3 = 338(44)\cdot10^{-6}K^{-1}$ and linear CTE: $\alpha_1 = -80(14)\cdot10^{-6}K^{-1}$

only the *b*-direction is common for the principal and the crystallographic axis

Determination of structural parameters characterising the short-range order in liquid crystalline phases of 7OS5

In the nematic phase, the correlation length $\xi_{||}$ in the direction parallel to the director was obtained by fitting an asymmetric Lorentz curve to the diffuse maximum at 2θ ≈ 3° (Figure S4(a)):

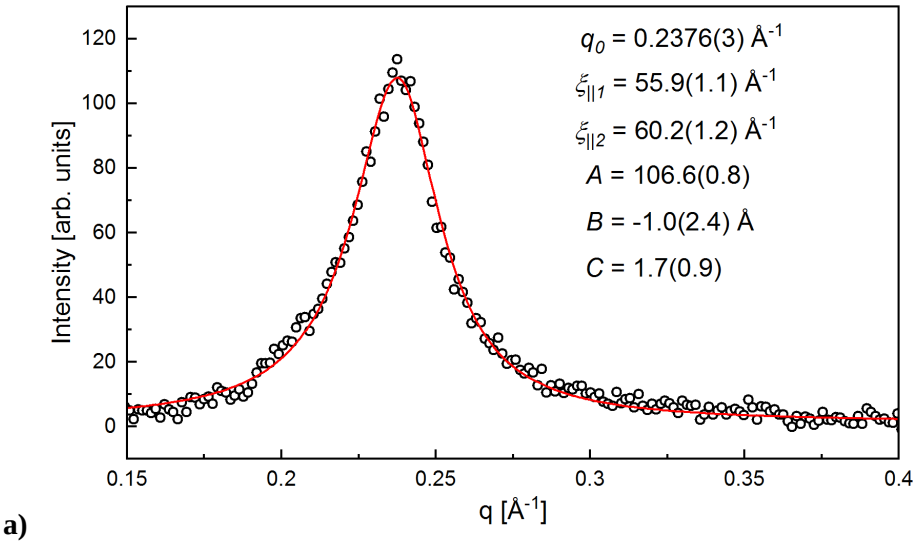
$$I(q) = \begin{cases} \frac{A}{1+\xi_{||1}^2(q-q_0)^2} + Bq + C \; for \; q < q_0 \\ \frac{A}{1+\xi_{||2}^2(q-q_0)^2} + Bq + C \; for \; q > q_0 \end{cases}, \quad \text{(S1)}$$

where $\xi_{||} = 2\xi_{||1}\xi_{||2}/(\xi_{||1} + \xi_{||2})$ is the harmonic mean of $\xi_{||1}$ and $\xi_{||2}$, A is the peak amplitude, q_0 is the position of the maximum, B and C are the parameters of the linear background.

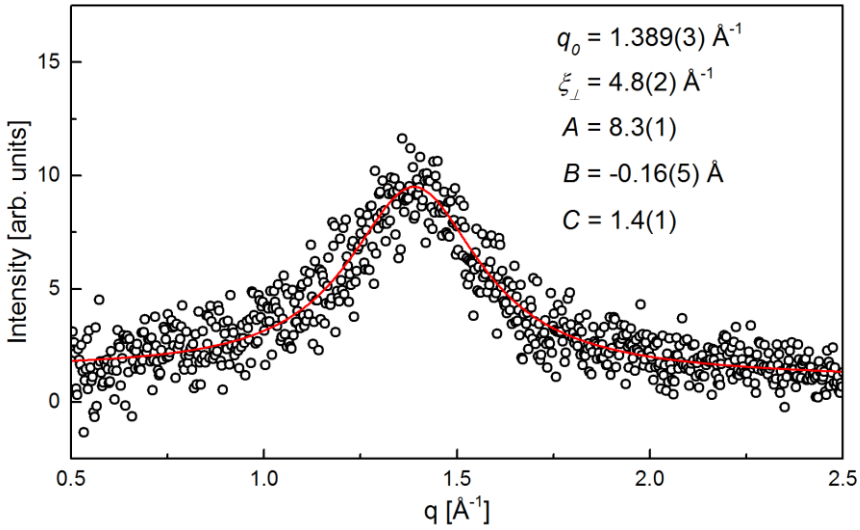
The correlation length $\xi_{\perp}$ in the direction perpendicular to the director was obtained by fitting the symmetric Lorentz curve to the diffuse maximum at 2θ ≈ 20° (Figure S4(b)):

$$I(q) = \frac{A}{1+\xi_{\perp}^2(q-q_0)^2} + Bq + C. \quad \text{(S2)}$$

The scattering vector values were calculated as $q = 4\pi sin\theta/\lambda_{CuK\alpha}$ using the weighted average $\lambda_{CuK\alpha} = \frac{2}{3}\lambda_{CuK\alpha1} + \frac{1}{3}\lambda_{CuK\alpha2}$.



a)



b)

Figure S4 Diffuse maxima visible in the powder diffraction patterns of the nematic phase of 7OS5 at q_0 = 0.24 Å^{-1} (a) and at q_0 = 1.4 Å^{-1} (b) with the fitting results of Equation S1 and Equation S2, respectively; results for 40°C during cooling.