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Structure of a Nematogenic Oxathiane Derivative

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Abstract. 4-Cyanophenyl *trans*-4-(*trans*-5-*n*-hexyl-1,3-oxathian-2-yl)cyclohexanoate, $C_{24}H_{33}NO_3S$, $M_r = 415.6$, triclinic, $P\bar{1}$, $a = 5.619$ (1), $b = 14.201$ (2), $c = 16.591$ (2) Å, $\alpha = 112.99$ (1), $\beta = 92.67$ (1), $\gamma = 104.08$ (1)°, $V = 1167.1$ (3) Å³, $Z = 2$, $D_x = 1.183$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 13.7$ cm⁻¹, $F(000) = 448$, $T = 291$ K, final $R = 0.054$ for 2647 observed reflections. The molecules adopt a rod-like shape, only very slightly 'banana' bent. The oxathiane ring has a distorted chair conformation and is most puckered in the vicinity of the O atom. The orientation of the oxathiane ring and the cyclohexane ring in the molecule is almost the same. Centrosymmetric pairs of molecules form a parallel imbricated packing within the crystal.

Experimental. Colourless, clear crystals were recrystallized from a methanol/benzene mixture. Phase behaviour: nematic range from 376 K (melting point) to 478 K (clearing point), appearance of a monotropic smectic A phase at 343 K by strong undercooling of the nematic phase. Synthesized by Tschierske, Joachimi, Vorbrod, Zschke, Wiegeleben, Hauser & Demus (1989). A crystal of dimensions 0.37 × 0.34 × 0.15 mm was mounted on a Syntex $P2_1$ diffractometer with graphite monochromator (angle 26.57°). Unit-cell parameters were determined by least-squares treatment of the setting angles for 13 counter reflections within $20 \leq 2\theta \leq 30^\circ$. Intensity data were collected to $2\theta = 115^\circ$, $-6 \leq h \leq 6$, $-15 \leq k \leq 15$, $0 \leq l \leq 18$, in $\theta/2\theta$ scan (bisecting mode). Intensity variation of two check reflections: $\pm 1.8\%$ for $04\bar{1}$ and $\pm 2.2\%$ for 025 . 3037 unique reflections, 2647 with $I \geq$

$1.96\sigma(I)$ were considered observed. Only L_p correction applied during data reduction. Structure solved by direct methods in *SHELXS86* (Sheldrick, 1986) and refined by full-matrix least-squares techniques on F in *SHELX76* (Sheldrick, 1976). The non-H atoms were refined with anisotropic displacement parameters, H atoms located in a difference Fourier map were refined isotropically in a separate block, resulting in a total of 6.7 reflections per parameter and a final R of 0.054. Unit weights were used throughout the refinement. $(\Delta/\sigma)_{\max} = 0.06$ in the last cycle, $\Delta\rho_{\max} = 0.30$ and $\Delta\rho_{\min} = -0.31$ e Å⁻³ in the final difference map. Atomic scattering factors were from *SHELX76*. All calculations were on an IBM XT computer. Final non-H-atom parameters are given in Table 1, selected bond lengths, angles and torsion angles in Table 2. The molecular structure with atom labels is shown in Fig. 1; Fig. 2 gives an impression of the packing arrangement in the crystalline phase of the mesogene.†

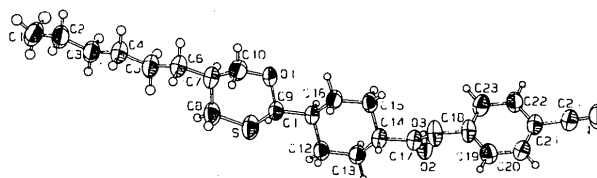
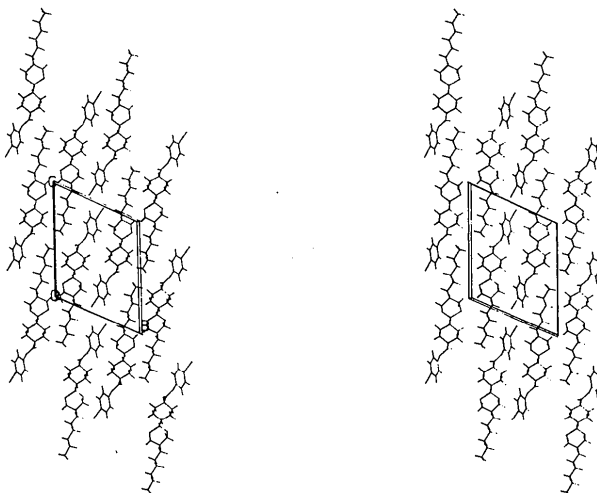
Related literature. To our knowledge only one crystal structure analysis of a 1,3-oxathiane derivative has been reported (2-*p*-nitrophenyl-1,3-oxathiane; de Wolf, Verschoor & Romers, 1972). The geometric parameters for the heterocycle agree well with those of the compound presented in this article. The mutual orientation of the two saturated rings is almost the same as that of the two cyclohexane moieties in other mesogenic bicyclohexyl derivatives

† Lists of structure factors, anisotropic displacement parameters for non-H atoms and H-atom parameters, as well as a fuller list of bond lengths and angles, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52937 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. Fractional coordinates of non-H atoms and equivalent isotropic displacement parameters (\AA^2)
$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
C1	-0.7656 (10)	0.0832 (4)	-0.3945 (3)	0.104 (2)
C2	-0.7516 (8)	0.1443 (3)	-0.2958 (2)	0.079 (2)
C3	-0.5763 (7)	0.1200 (3)	-0.2399 (2)	0.072 (2)
C4	-0.5462 (7)	0.1856 (3)	-0.1411 (2)	0.071 (2)
C5	-0.3623 (7)	0.1633 (3)	-0.0867 (2)	0.073 (2)
C6	-0.3281 (7)	0.2311 (3)	0.0122 (2)	0.071 (2)
C7	-0.1286 (6)	0.2161 (3)	0.0680 (2)	0.062 (2)
C8	-0.1981 (7)	0.1090 (3)	0.0720 (2)	0.076 (2)
C9	0.0731 (6)	0.2132 (3)	0.2374 (2)	0.055 (2)
C10	-0.0811 (7)	0.3035 (3)	0.1623 (2)	0.070 (2)
C11	0.2888 (6)	0.2279 (3)	0.3047 (2)	0.050 (1)
C12	0.2518 (6)	0.1364 (3)	0.3325 (2)	0.061 (2)
C13	0.4685 (6)	0.1513 (3)	0.3992 (2)	0.060 (2)
C14	0.5191 (6)	0.2578 (3)	0.4804 (2)	0.052 (2)
C15	0.5532 (6)	0.3501 (3)	0.4530 (2)	0.063 (2)
C16	0.3300 (6)	0.3328 (3)	0.3873 (2)	0.060 (2)
C17	0.7468 (6)	0.2757 (3)	0.5425 (2)	0.056 (2)
C18	0.9634 (6)	0.3904 (3)	0.6889 (2)	0.059 (2)
C19	0.9280 (6)	0.3759 (3)	0.7648 (2)	0.063 (2)
C20	1.1230 (7)	0.4175 (3)	0.8339 (2)	0.062 (2)
C21	1.3523 (6)	0.4731 (3)	0.8252 (2)	0.055 (2)
C22	1.3869 (6)	0.4865 (3)	0.7478 (2)	0.064 (2)
C23	1.1906 (7)	0.4452 (3)	0.6789 (2)	0.067 (2)
C24	1.5594 (7)	0.5201 (3)	0.8974 (2)	0.063 (2)
N	1.7244 (6)	0.5576 (3)	0.9533 (2)	0.085 (2)
O1	0.1194 (4)	0.3043 (2)	0.2180 (1)	0.067 (1)
O2	0.9049 (5)	0.2328 (2)	0.5269 (2)	0.084 (1)
O3	0.7538 (4)	0.3554 (2)	0.6239 (1)	0.070 (1)
S	0.0370 (2)	0.09148 (8)	0.13846 (6)	0.0784 (5)

Fig. 1. ORTEP plot (Johnson, 1976) of the molecular structure with atom-labelling scheme. (Ellipsoids are at the 50% probability level, H atoms are at an arbitrary scale corresponding to their U_{iso} values.)Fig. 2. Stereo PLUTO drawing (Motherwell & Clegg, 1978) of the molecular packing viewed down the a axis.Table 2. Selected bond lengths (\AA), angles and torsion angles ($^\circ$)

C6—C7	1.527 (6)	C10—C7	1.530 (4)
C7—C8	1.505 (6)	C9—C11	1.525 (5)
C8—S	1.805 (5)	C14—C17	1.509 (5)
S—C9	1.817 (3)	C17—O2	1.178 (5)
C9—O1	1.420 (5)	C17—O3	1.374 (4)
O1—C10	1.418 (5)	O3—C18	1.405 (4)
C6—C7—C8	113.4 (4)	C9—C11—C12	112.9 (3)
C6—C7—C10	108.7 (4)	C9—C11—C16	110.5 (4)
C10—C7—C8	108.9 (3)	C13—C14—C17	110.6 (3)
C7—C8—S	113.4 (3)	C15—C14—C17	109.7 (3)
C8—S—C9	97.2 (2)	C14—C17—O2	127.9 (3)
S—C9—O1	111.8 (2)	C14—C17—O3	109.9 (4)
C9—O1—C10	113.6 (3)	O2—C17—O3	122.2 (4)
O1—C10—C7	113.7 (4)	C17—O3—C18	118.5 (4)
O1—C9—C11	108.9 (3)	O3—C18—C19	117.4 (4)
S—C9—C11	109.6 (3)	O3—C18—C23	120.6 (3)
C5—C6—C7—C10	169.3 (4)	C10—C7—C8—S	-58.6 (5)
C6—C7—C10—O1	-173.8 (4)	C8—S—C9—C11	-173.5 (3)
C7—C8—S—C9	52.0 (4)	S—C9—C11—C16	177.6 (3)
C8—S—C9—O1	-52.6 (4)	C15—C14—C17—O3	72.7 (5)
S—C9—O1—C10	64.4 (4)	C14—C17—O3—C18	-174.6 (4)
C9—O1—C10—C7	-67.1 (5)	C17—O3—C18—C23	68.3 (6)
O1—C10—C7—C8	62.2 (5)		

investigated earlier (Krieg, Deutscher, Baumeister, Hartung & Jaskólski, 1989, and references therein). The slightly 'banana-bent' overall molecular shape is caused by the phenyl cyclohexanoate part of the compound as described by Baumeister, Brandt, Hartung, Wedler, Deutscher, Frach & Jaskólski (1985) for two mesogenic phenyl cyclohexanoate derivatives.

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