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Structure of 9-Fluorenyl *O*-Palmitoyl Ketone Oxime

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Abstract. 9-Fluorenylideneamino palmitate $C_{29}H_{39}NO_2$, $M_r = 433\cdot64$, orthorhombic, $P2_12_12_1$, $a = 60\cdot27$ (1), $b = 7\cdot574$ (2), $c = 5\cdot588$ (2) Å, $V = 2551$ (1) Å^3, $Z = 4$, D_m(flotation) = 1·12, $D_x = 1\cdot129$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 5\cdot17$ cm^{-1}, $F(000) = 944$, $T = 295$ K, $R = 0\cdot047$ for 2110 unique reflections. The C=N—O—C(=O)—C linkage of the oxime part has a *trans* zigzag form. The fluorenyl group is slightly non-planar, and the palmitoyl chain is also slightly distorted. The molecules are packed in bilayers with an orthorhombic subcell arrangement of the alkyl chains.

Introduction. The structure of the title compound was determined as part of a study of the molecular packing of long chain molecules. In a previous paper (Taga & Miyasaka, 1987), we reported two different types of conformers for similar compounds, *O*-palmitoyl benzophenone oxime and (*E*)-*O*-palmitoyl phenyl 2-pyridyl ketone oxime. The conformations of these molecules were different at the oxime groups, and the calculated dipole moments of these groups were quite different. The aromatic rings at the head groups of both molecules have different tilt angles with respect to their oxime planes. Thus some relationships between the tilt angle of the aromatic ring and the conformation of the oxime part were supposed. The title compound has an approximately planar fluorenyl group, and corresponds to the case of non-tilting aromatic rings. A slightly non-planar fluorene structure was reported recently (Gerkin, Lundstedt & Reppart, 1984). The planarity of the fluorenyl group was also of interest in this structure.

Experimental. Colourless transparent crystal from ether, 0·2 × 0·2 × 0·3 mm; Rigaku AFC-5RU diffractometer with graphite-monochromated Cu $K\alpha$ radiation; unit-cell parameters from 20 reflections with $44 < 2\theta < 55°$; 4058 reflections measured within $2\theta < 120°$ (h −66→66, k 0→8, l 0→6); ω–2θ scan at 4° min^{-1}; three standard reflections monitored every 56 reflections, no significant change in intensities; 2110 unique reflections with $F_o > 3\sigma(F_o)$, $R_{\rm int} = 0\cdot048$; no absorption corrections; structure solved by direct methods using *RANTAN* (Yao Jia-Xing, 1983); refined by full-matrix least squares, $\sum w(|F_o| - |F_c|)^2$ minimized, $w = [\sigma^2(F_o) + (0\cdot023F_o)]^{-1}$; non-H atoms anisotropic; H atoms from D map isotropic; $R = 0\cdot047$, $wR = 0\cdot065$, $S = 1\cdot67$, $(\Delta/\sigma)_{\rm max} = 0\cdot88$; $\Delta\rho_{\rm max} = 0\cdot13$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Computations performed on a FACOM M382 in the Data Processing Center of Kyoto University, using *KPPXRAY* programs (Taga, Higashi & Iizuka, 1985).

Discussion. The final atomic parameters are listed in Table 1.* Bond distances and angles are listed in Table 2.

Fig. 1 shows a perspective view of the molecule. The conformation of the oxime part is the *O*-palmitoyl benzophenone oxime type. That is, the C(2)—N(1)—O(15)—C(16)—C(18) bond has a *trans* zigzag form; the torsion angles are −175·5 (2)° for C(2)—N(1)—O(15)—C(16) and −174·7 (2)° for N(1)—O(15)—C(16)—C(18). The fluorene group is approximately planar, but the two six-membered rings do not lie exactly on the same plane. They are related by an approximate mirror plane passing through C(2) and bisecting the C(8)—C(14) bond, and the dihedral angle between the two least-squares planes is 4·0°. Similar non-planar fluorene was observed at 159 K (Gerkin, Lundstedt & Reppart, 1984).

The O—C=O plane tilts from these planes with the dihedral angles 9·2 and 13·2°, and the non-bonding distance between C(10) and O(15) is 2·898 (4) Å; this conformation reduces the repulsion between these atoms. The alkyl chain with an extended form is slightly bent near the head group. The C(18)—C(19) bond

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* Lists of atomic parameters for H atoms, anisotropic thermal parameters for non-H atoms and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51302 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Atomic parameters for non-hydrogen atoms*

$$B_{\text{eq}} = \tfrac{4}{3}\textstyle\sum_i\sum_j B_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
N(1)	0·31660 (3)	0·3859 (3)	0·7111 (5)	4·07 (5)
C(2)	0·30730 (4)	0·4636 (4)	0·8897 (5)	3·61 (5)
C(3)	0·28910 (4)	0·3765 (4)	1·0201 (5)	3·56 (6)
C(4)	0·27832 (5)	0·2168 (4)	0·9786 (6)	4·44 (6)
C(5)	0·26191 (5)	0·1661 (4)	1·1365 (6)	4·70 (7)
C(6)	0·25638 (5)	0·2685 (4)	1·3333 (6)	4·58 (7)
C(7)	0·26728 (5)	0·4287 (4)	1·3757 (5)	4·24 (6)
C(8)	0·28348 (4)	0·4800 (3)	1·2167 (5)	3·51 (5)
C(9)	0·31135 (4)	0·6346 (3)	1·0103 (5)	3·69 (6)
C(10)	0·32533 (5)	0·7754 (4)	0·9589 (6)	4·54 (6)
C(11)	0·32510 (5)	0·9196 (4)	1·1088 (7)	5·07 (7)
C(12)	0·31173 (5)	0·9256 (4)	1·3102 (6)	4·78 (7)
C(13)	0·29750 (5)	0·7867 (4)	1·3616 (5)	4·48 (6)
C(14)	0·29729 (4)	0·6429 (4)	1·2113 (5)	3·55 (5)
O(15)	0·33460 (3)	0·4916 (3)	0·6259 (4)	4·57 (4)
C(16)	0·34445 (4)	0·4263 (4)	0·4268 (5)	3·78 (6)
O(17)	0·33787 (4)	0·3025 (3)	0·3175 (4)	5·88 (6)
C(18)	0·36436 (5)	0·5375 (4)	0·3663 (6)	4·79 (6)
C(19)	0·37762 (5)	0·4673 (4)	0·1601 (6)	4·97 (7)
C(20)	0·39977 (5)	0·5585 (4)	0·1311 (6)	5·01 (7)
C(21)	0·41409 (5)	0·4812 (4)	−0·0651 (6)	5·01 (7)
C(22)	0·43716 (5)	0·5620 (4)	−0·0821 (6)	5·02 (7)
C(23)	0·45132 (5)	0·4844 (4)	−0·2795 (6)	4·88 (7)
C(24)	0·47461 (5)	0·5631 (5)	−0·2942 (6)	4·98 (7)
C(25)	0·48865 (5)	0·4869 (4)	−0·4939 (6)	5·08 (7)
C(26)	0·51187 (5)	0·5639 (5)	−0·5101 (6)	4·95 (6)
C(27)	0·52592 (5)	0·4854 (5)	−0·7068 (6)	5·14 (7)
C(28)	0·54907 (5)	0·5614 (5)	−0·7260 (6)	5·08 (7)
C(29)	0·56293 (5)	0·4803 (4)	−0·9242 (6)	5·06 (7)
C(30)	0·58638 (5)	0·5528 (5)	−0·9438 (7)	5·30 (8)
C(31)	0·59976 (5)	0·4717 (5)	−1·1445 (7)	5·86 (9)
C(32)	0·62318 (6)	0·5411 (6)	−1·1636 (9)	8·02 (10)

Table 2. *Bond lengths* (Å) *and angles* (°)

N(1)—C(2)	1·287 (4)	N(1)—O(15)	1·430 (3)
C(2)—C(3)	1·473 (4)	C(2)—C(9)	1·480 (4)
C(3)—C(4)	1·392 (4)	C(3)—C(8)	1·391 (4)
C(4)—C(5)	1·380 (4)	C(5)—C(6)	1·386 (5)
C(6)—C(7)	1·400 (4)	C(7)—C(8)	1·376 (4)
C(8)—C(14)	1·489 (4)	C(9)—C(10)	1·389 (4)
C(9)—C(14)	1·408 (4)	C(10)—C(11)	1·376 (5)
C(11)—C(12)	1·385 (5)	C(12)—C(13)	1·387 (4)
C(13)—C(14)	1·375 (4)	O(15)—C(16)	1·355 (4)
C(16)—O(17)	1·187 (4)	C(16)—C(18)	1·505 (4)
C(18)—C(19)	1·500 (5)	C(19)—C(20)	1·512 (4)
C(20)—C(21)	1·513 (5)	C(21)—C(22)	1·522 (4)
C(22)—C(23)	1·513 (5)	C(23)—C(24)	1·527 (4)
C(24)—C(25)	1·515 (5)	C(25)—C(26)	1·519 (4)
C(26)—C(27)	1·510 (5)	C(27)—C(28)	1·513 (4)
C(28)—C(29)	1·517 (5)	C(29)—C(30)	1·520 (4)
C(30)—C(31)	1·512 (5)	C(31)—C(32)	1·510 (5)

C(2)—N(1)—O(15)	109·4 (2)	N(1)—C(2)—C(3)	120·2 (3)
N(1)—C(2)—C(9)	132·9 (2)	C(3)—C(2)—C(9)	106·8 (2)
C(2)—C(3)—C(4)	130·9 (3)	C(2)—C(3)—C(8)	108·6 (2)
C(4)—C(3)—C(8)	120·5 (3)	C(3)—C(4)—C(5)	118·0 (3)
C(4)—C(5)—C(6)	121·6 (3)	C(5)—C(6)—C(7)	120·4 (3)
C(6)—C(7)—C(8)	117·9 (3)	C(3)—C(8)—C(7)	121·6 (2)
C(3)—C(8)—C(14)	108·3 (2)	C(7)—C(8)—C(14)	130·1 (2)
C(2)—C(9)—C(10)	132·7 (3)	C(2)—C(9)—C(14)	107·6 (2)
C(10)—C(9)—C(14)	119·7 (3)	C(9)—C(10)—C(11)	118·5 (3)
C(10)—C(11)—C(12)	121·8 (3)	C(11)—C(12)—C(13)	120·2 (3)
C(12)—C(13)—C(14)	118·7 (3)	C(8)—C(14)—C(9)	108·4 (2)
C(8)—C(14)—C(13)	130·5 (2)	C(9)—C(14)—C(13)	121·1 (3)
N(1)—O(15)—C(16)	113·7 (2)	O(15)—C(16)—O(17)	124·4 (3)
O(15)—C(16)—C(18)	109·3 (2)	O(17)—C(16)—C(18)	126·4 (3)
C(16)—C(18)—C(19)	113·5 (3)	C(18)—C(19)—C(20)	113·0 (3)
C(19)—C(20)—C(21)	113·9 (3)	C(20)—C(21)—C(22)	114·2 (3)
C(21)—C(22)—C(23)	113·9 (3)	C(22)—C(23)—C(24)	113·9 (3)
C(23)—C(24)—C(25)	113·9 (3)	C(24)—C(25)—C(26)	114·4 (3)
C(25)—C(26)—C(27)	114·1 (3)	C(26)—C(27)—C(28)	114·8 (3)
C(27)—C(28)—C(29)	113·9 (3)	C(28)—C(29)—C(30)	114·7 (3)
C(29)—C(30)—C(31)	113·7 (3)	C(30)—C(31)—C(32)	114·2 (3)

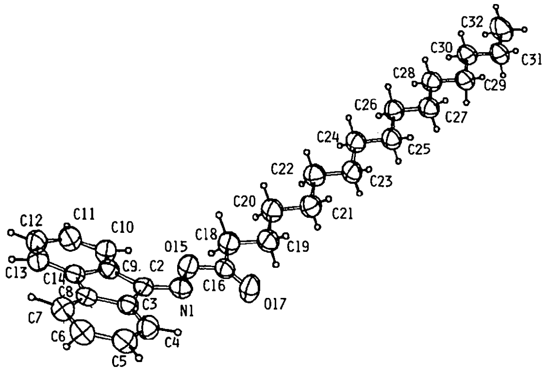

Fig. 1. A perspective view of the molecule with atomic numbering.

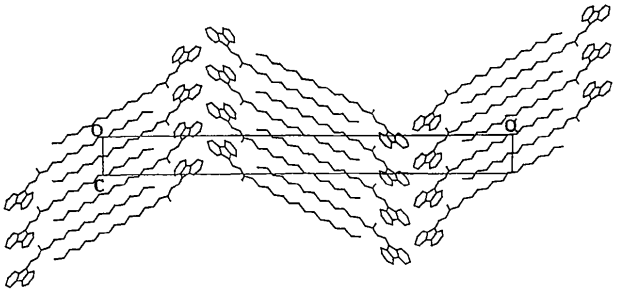

Fig. 2. Packing of the molecules viewed down **b**.

distance of 1·498 (4) Å is significantly shorter than the other C—C distances of the alkyl chain, and the C(16)—C(18)—C(19)—C(20) torsion angle of −168·9 (2)° deviates greatly from the normal *trans* angle (180°). Thus the chain distortion mainly occurs around the C(18)—C(19) bond.

Fig. 2 shows the molecular packing projected along the *b* axis. The molecules are packed in bilayers with thickness 30·1 Å and inclination angle 28·5° for the long chain axis from the (100) layer surface. The palmitoyl chains are packed in an orthorhombic subcell form with dimensions $a_s = 4{\cdot}92$ (3), $b_s = 7{\cdot}574$ (2) and $c_s = 2{\cdot}545$ (6) Å. This chain packing is the usual form observed in polyethylene or *n*-paraffin (Abrahamsson, Dahlen, Lofgren & Pascher, 1978). The short intermolecular contacts are 3·220 (4) Å for O(17)···C(11) (+*x*, 1+*y*, 1+*z*) and 3·257 (3) Å for O(17)···C(2) (+*x*, +*y*, 1+*z*) between the head groups in the same layer. The chain distortion is affected by the molecular packing.

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