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Ethyl and Butyl 4-[4-(4-*n*-Octyloxybenzoyloxy)benzylidene]aminobenzoates

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Abstract

In order to clarify relationships between the mesophase behaviour and crystal structures, the X-ray structure determinations of the title compounds, ethyl 4-[4-(4-*n*-octyloxybenzoyloxy)benzylidene]aminobenzoate, $C_{31}H_{35}NO_5$, and butyl 4-[4-(4-*n*-octyloxybenzoyloxy)benzylidene]aminobenzoate, $C_{33}H_{39}NO_5$, have been performed. The compounds have different phase sequences, *i.e.* crystal–smectic A–nematic–isotropic and crystal–smectic C–smectic A–nematic–isotropic for the ethyl and butyl compounds, respectively.

Comment

The homologous series of *n*-alkyl 4-[4-(4-*n*-octyloxybenzoyloxy)benzylidene]aminobenzoates have the phase sequence crystal–smectic A–nematic–isotropic for $n \leq 3$ and crystal–smectic C–smectic A–nematic–isotropic for $n \geq 4$ (Lotke & Patil, 1989).

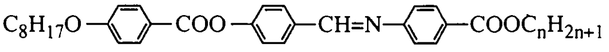

Fig. 1 shows the molecular structures and numbering schemes of both compounds. The butyl compound has two crystallographically independent molecules, *A* and *B*. In all three molecules, *i.e.* the single molecule of the ethyl compound and the *A* and *B* molecules of the butyl compound, the octyloxy chains have all-*trans* conformations. The core moieties, including the three phenylene rings, also have very similar conformations. The phenyl rings C1–C6 and C15–C20 are nearly coplanar, with dihedral angles of 1.7 (4), 2.1 (1) and 3.69 (7)° for $n = 2$, $n = 4$ (molecule *A*) and $n = 4$ (molecule *B*), respectively. The central ring, C8–C13, is twisted with respect to both of the terminal rings. The dihedral angles with rings C1–C6 and C15–C20 are 59.7 (2) and 60.5 (2)°, respectively, for $n = 2$, 54.0 (1) and 54.4 (1)° for $n = 4$ (molecule *A*), and 56.8 (1) and 54.3 (1)° for $n = 4$ (molecule *B*). The C21—O1—C22—C23 torsion angles are 172.9 (9), 179.3 (4) and 118.4 (6)° for $n = 2$, $n = 4$ (molecule *A*) and $n = 4$ (molecule *B*), respectively. The terminal atoms of the butyl chains are disordered in both molecules of the butyl compound.

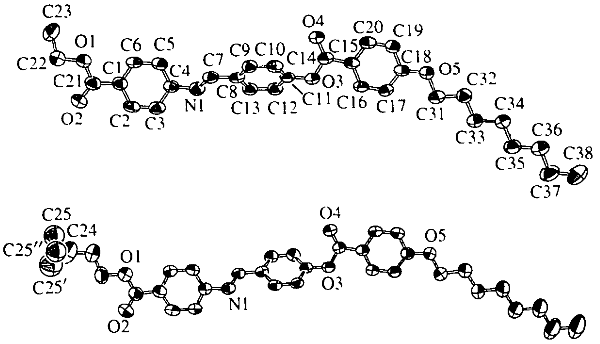

Fig. 1. The molecular structures of the ethyl (upper) and butyl (molecule *A*) compounds, with 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

Figs. 2 and 3 show the crystal structures of the ethyl and butyl compounds, respectively. Both crystals have smectic-like layer structures, with the layers parallel to the *bc* plane in the ethyl compound and parallel to the *ac* plane in the butyl compound. In the layers, adjacent molecules are arranged alternately so as to cancel their longitudinal dipole moments with one another. The core moieties overlap to a great extent in both crystals. The nearest neighboring phenyl rings of adjacent molecules are at angles of about 60°. In the ethyl compound, the core moieties are almost perpendicular to the layer plane, with a tilt angle of 7°, while in the butyl compound, these moieties are tilted to a greater extent (by about 20°) in the layer. These features correspond to the fact that the former compound transforms to the smectic A structure, in which molecular long axes are upright in a layer, while the latter compound transforms to the smectic C structure, in which molecular long axes are tilted.

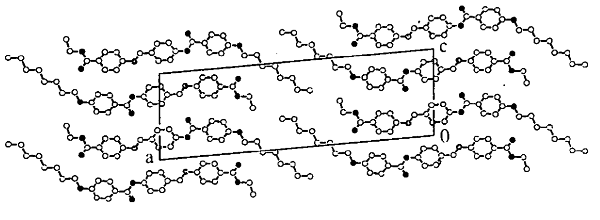

Fig. 2. The crystal structure of the ethyl compound viewed along the b axis. Open, closed and hatched circles denote C, O and N atoms, respectively.

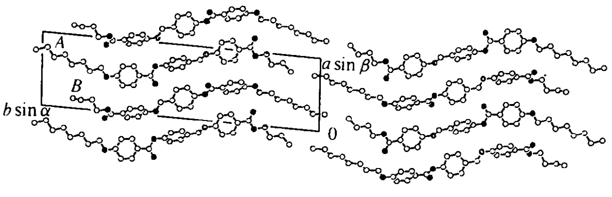

Fig. 3. The crystal structure of the butyl compound viewed along the c axis. Only a half unit cell along the c axis is shown for clarity. Inversion-related molecules are overlapped in the full unit cell. Disordered atoms with minor occupancies are omitted for simplicity.

Experimental

Both title compounds were synthesized according to the conventional method of Lotke & Patil (1989) and crystallized from toluene solution.

Ethyl compound ($n = 2$)

Crystal data

$C_{31}H_{35}NO_5$
$M_r = 501.60$
Monoclinic
$P2_1$
$a = 32.686\ (4)$ Å
$b = 4.094\ (5)$ Å
$c = 10.257\ (7)$ Å
$\beta = 95.08\ (3)°$
$V = 1367\ (2)$ Å^3
$Z = 2$
$D_x = 1.219$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 18 reflections
$\theta = 20.0$–$26.5°$
$\mu = 0.659$ mm^{-1}
$T = 293\ (2)$ K
Plate
0.60 × 0.40 × 0.05 mm
Colourless

Data collection

Rigaku AFC-7R four-circle diffractometer
$2\theta/\omega$ scans
Absorption correction: none
2522 measured reflections
2373 independent reflections
1718 observed reflections $[I > 2\sigma(I)]$

$R_{\rm int} = 0.08$
$\theta_{\rm max} = 59.94°$
$h = -36 \rightarrow 36$
$k = -4 \rightarrow 0$
$l = 0 \rightarrow 11$
3 standard reflections monitored every 150 reflections
intensity decay: 0.82%

Refinement

Refinement on F^2
$R(F) = 0.0692$
$wR(F^2) = 0.8720$
$S = 1.052$
2338 reflections
302 parameters
$w = 1/[\sigma^2(F_o^2) + (0.1545P)^2 + 0.5758P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.409$

$\Delta\rho_{\rm max} = 0.249$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.246$ e Å^{-3}
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983) parameter = 0.16 (75)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for the ethyl compound*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
O1	0.71637 (13)	0.0898 (15)	0.6589 (4)	0.075 (2)
O2	0.71324 (14)	0.290 (2)	0.8585 (4)	0.099 (2)
O3	1.10040 (12)	0.2386 (15)	0.7638 (3)	0.0695 (15)
O4	1.10336 (13)	−0.0020 (17)	0.5680 (4)	0.083 (2)
O5	1.29205 (12)	0.1670 (14)	0.7583 (4)	0.0664 (12)
N1	0.9077 (2)	0.1318 (18)	0.8338 (4)	0.068 (2)
C1	0.77902 (9)	0.1654 (11)	0.7860 (3)	0.059 (2)
C2	0.80041 (11)	0.3013 (11)	0.8960 (3)	0.066 (2)
C3	0.84306 (11)	0.2887 (13)	0.9106 (3)	0.067 (2)
C4	0.86434 (9)	0.1402 (15)	0.8152 (3)	0.058 (2)
C5	0.84295 (11)	0.0043 (13)	0.7051 (3)	0.063 (2)
C6	0.80030 (11)	0.0169 (11)	0.6905 (3)	0.059 (2)
C7	0.9284 (2)	0.237 (2)	0.7436 (5)	0.061 (2)
C8	0.97282 (9)	0.2307 (12)	0.7496 (3)	0.060 (2)
C9	0.99172 (11)	0.3823 (12)	0.6495 (3)	0.063 (2)
C10	1.03430 (11)	0.3833 (13)	0.6516 (3)	0.065 (2)
C11	1.05798 (9)	0.2327 (14)	0.7537 (3)	0.061 (2)
C12	1.03908 (11)	0.0811 (14)	0.8537 (3)	0.067 (2)
C13	0.99650 (11)	0.0801 (12)	0.8517 (3)	0.063 (2)
C14	1.1204 (2)	0.1236 (19)	0.6651 (5)	0.062 (2)
C15	1.16545 (9)	0.1512 (11)	0.6915 (3)	0.056 (2)
C16	1.18325 (11)	0.3020 (11)	0.8040 (3)	0.058 (2)
C17	1.22575 (11)	0.3131 (13)	0.8280 (3)	0.061 (2)
C18	1.25045 (9)	0.1735 (14)	0.7395 (3)	0.056 (2)
C19	1.23266 (11)	0.0227 (13)	0.6270 (3)	0.064 (2)
C20	1.19015 (11)	0.0116 (11)	0.6030 (3)	0.065 (2)
C21	0.7339 (2)	0.191 (2)	0.7749 (6)	0.069 (2)
C22	0.6726 (2)	0.087 (3)	0.6414 (7)	0.089 (3)
C23	0.6597 (2)	0.008 (4)	0.5010 (8)	0.116 (4)
C31	1.3110 (2)	0.291 (2)	0.8795 (5)	0.070 (2)
C32	1.3561 (2)	0.247 (2)	0.8844 (5)	0.066 (2)
C33	1.3765 (2)	0.327 (2)	1.0203 (6)	0.071 (2)
C34	1.4226 (2)	0.301 (2)	1.0353 (6)	0.071 (2)
C35	1.4413 (2)	0.374 (3)	1.1733 (6)	0.078 (2)
C36	1.4877 (2)	0.355 (3)	1.1906 (6)	0.083 (2)
C37	1.5058 (3)	0.431 (3)	1.3291 (7)	0.103 (3)
C38	1.5515 (3)	0.403 (5)	1.3486 (9)	0.148 (5)

Table 2. *Selected geometric parameters (Å, °) for the ethyl compound*

O1—C21	1.339 (7)	O5—C31	1.432 (7)
O1—C22	1.425 (8)	N1—C7	1.268 (8)
O2—C21	1.210 (7)	N1—C4	1.414 (6)
O3—C14	1.338 (7)	C1—C21	1.471 (7)
O3—C11	1.381 (5)	C8—C7	1.448 (7)
O4—C14	1.212 (7)	C15—C14	1.478 (7)
O5—C18	1.356 (5)		
C7—N1—C4	119.1 (5)	N1—C7—C8	124.1 (5)

Butyl compound (n = 4)

Crystal data

$C_{33}H_{39}NO_5$
$M_r = 529.65$
Triclinic
$P\overline{1}$
$a = 9.577$ (2) Å
$b = 34.681$ (6) Å
$c = 9.562$ (3) Å
$\alpha = 97.43$ (2)°
$\beta = 108.28$ (2)°
$\gamma = 83.74$ (2)°
$V = 2982.5$ (12) $Å^3$
$Z = 4$
$D_x = 1.180$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 21 reflections
$\theta = 23.0–25.5°$
$\mu = 0.628$ mm^{-1}
$T = 293$ (2) K
Plate
0.50 × 0.40 × 0.10 mm
Colourless

Data collection

Rigaku AFC-7*R* four-circle diffractometer
$2\theta/\omega$ scans
Absorption correction: none
9472 measured reflections
8857 independent reflections
6314 observed reflections $[I > 2\sigma(I)]$

$R_{\rm int} = 0.01$
$\theta_{\rm max} = 60.01°$
$h = 0 \rightarrow 10$
$k = -38 \rightarrow 38$
$l = -10 \rightarrow 10$
3 standard reflections monitored every 150 reflections intensity decay: 0.18%

Refinement

Refinement on F^2
$R(F) = 0.0725$
$wR(F^2) = 0.5923$
$S = 1.066$
8785 reflections
640 parameters
$w = 1/[\sigma^2(F_o^2) + (0.1378P)^2 + 1.4509P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = -0.132$
$\Delta\rho_{\rm max} = 0.482$ e $Å^{-3}$
$\Delta\rho_{\rm min} = -0.342$ e $Å^{-3}$
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$) for the butyl compound*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}/U_{\rm iso}$
O1*A*	0.9476 (3)	1.23387 (7)	2.0070 (3)	0.0877 (7)
O2*A*	1.1809 (3)	1.23838 (8)	2.1440 (3)	0.1026 (9)
O3*A*	0.6740 (2)	1.59218 (6)	2.3011 (2)	0.0641 (5)
O4*A*	0.4337 (2)	1.59085 (7)	2.1695 (2)	0.0720 (6)
O5*A*	0.4638 (2)	1.76821 (6)	2.4290 (3)	0.0732 (6)
N1*A*	0.9032 (3)	1.41129 (8)	2.2711 (3)	0.0618 (6)
C1*A*	1.0109 (2)	1.29408 (4)	2.1438 (2)	0.0611 (8)
C2*A*	0.8645 (2)	1.30873 (5)	2.0992 (2)	0.0635 (8)
C3*A*	0.82703 (15)	1.34749 (6)	2.1396 (2)	0.0624 (8)
C4*A*	0.9360 (2)	1.37160 (4)	2.2246 (2)	0.0558 (7)
C5*A*	1.0825 (2)	1.35696 (5)	2.2691 (2)	0.0642 (8)
C6*A*	1.11990 (15)	1.31820 (6)	2.2287 (2)	0.0661 (8)
C7*A*	0.8207 (3)	1.43273 (10)	2.1766 (3)	0.0572 (8)
C8*A*	0.7790 (2)	1.47384 (4)	2.2120 (2)	0.0528 (7)
C9*A*	0.7000 (2)	1.49535 (5)	2.09551 (14)	0.0593 (8)
C10*A*	0.6619 (2)	1.53479 (5)	2.1232 (2)	0.0640 (8)
C11*A*	0.7028 (2)	1.55271 (4)	2.2673 (2)	0.0557 (7)
C12*A*	0.7818 (2)	1.53120 (5)	2.38378 (14)	0.0616 (8)
C13*A*	0.8199 (2)	1.49176 (5)	2.3561 (2)	0.0601 (8)
C14*A*	0.5351 (3)	1.60918 (9)	2.2456 (3)	0.0557 (7)
C15*A*	0.5250 (2)	1.65102 (4)	2.2919 (2)	0.0535 (7)
C16*A*	0.3858 (2)	1.67063 (5)	2.2557 (2)	0.0609 (8)
C17*A*	0.36951 (15)	1.71013 (5)	2.3012 (2)	0.0628 (8)
C18*A*	0.4924 (2)	1.73002 (4)	2.3830 (2)	0.0592 (7)
C19*A*	0.6315 (2)	1.71041 (5)	2.4192 (2)	0.0620 (8)
C20*A*	0.64781 (14)	1.67091 (5)	2.3737 (2)	0.0577 (7)
C21*A*	1.0576 (4)	1.25339 (11)	2.1013 (4)	0.0716 (9)
C22*A*	0.9873 (5)	1.19452 (12)	1.9550 (5)	0.0982 (13)
C23*A*	0.8541 (6)	1.17688 (15)	1.8538 (7)	0.138 (2)
C24*A*	0.8925 (10)	1.1364 (2)	1.7789 (11)	0.206 (4)
C25*A*†	0.7810 (17)	1.1076 (5)	1.739 (2)	0.165 (6)‡
C25*A*′†	0.964 (4)	1.1033 (7)	1.861 (3)	0.209 (13)‡
C25*A*′′†	0.856 (3)	1.1074 (7)	1.858 (3)	0.159 (8)‡
C31*A*	0.5846 (4)	1.78864 (10)	2.5282 (4)	0.0754 (9)
C32*A*	0.5248 (4)	1.82785 (11)	2.5791 (5)	0.0844 (11)
C33*A*	0.6459 (4)	1.85254 (11)	2.6738 (5)	0.0897 (11)
C34*A*	0.5933 (5)	1.89299 (11)	2.7293 (5)	0.0988 (13)
C35*A*	0.7178 (5)	1.91768 (13)	2.8194 (6)	0.115 (2)
C36*A*	0.6692 (6)	1.95851 (14)	2.8711 (7)	0.131 (2)
C37*A*	0.7967 (8)	1.9827 (2)	2.9566 (9)	0.178 (3)
C38*A*	0.7477 (9)	2.0236 (2)	3.0032 (10)	0.231 (4)
O1*B*	0.9783 (4)	0.24027 (8)	0.4656 (3)	0.1087 (10)
O2*B*	1.0973 (4)	0.22906 (9)	0.6969 (4)	0.1187 (11)
O3*B*	0.6437 (2)	0.58773 (6)	0.8184 (2)	0.0626 (5)
O4*B*	0.5235 (3)	0.59797 (7)	0.5822 (2)	0.0806 (7)
O5*B*	0.4655 (3)	0.76666 (7)	0.9087 (3)	0.0821 (7)
N1*B*	0.9389 (3)	0.41354 (8)	0.7631 (3)	0.0637 (7)
C1*B*	1.0086 (2)	0.29379 (4)	0.6446 (2)	0.0596 (7)
C2*B*	0.9406 (2)	0.31837 (6)	0.5349 (2)	0.0638 (8)
C3*B*	0.9183 (2)	0.35811 (5)	0.5729 (2)	0.0630 (8)
C4*B*	0.9640 (2)	0.37327 (4)	0.7206 (2)	0.0559 (7)
C5*B*	1.0320 (2)	0.34869 (6)	0.83028 (15)	0.0679 (9)
C6*B*	1.0543 (2)	0.30895 (6)	0.7923 (2)	0.0699 (9)
C7*B*	0.8123 (3)	0.42984 (10)	0.7015 (3)	0.0561 (7)
C8*B*	0.7719 (2)	0.47107 (4)	0.7280 (2)	0.0513 (7)
C9*B*	0.6239 (2)	0.48384 (5)	0.6757 (2)	0.0542 (7)
C10*B*	0.57949 (14)	0.52291 (5)	0.7018 (2)	0.0561 (7)
C11*B*	0.6831 (2)	0.54920 (4)	0.7801 (2)	0.0531 (7)
C12*B*	0.8312 (2)	0.53643 (5)	0.8324 (2)	0.0599 (8)
C13*B*	0.87558 (13)	0.49736 (5)	0.8063 (2)	0.0596 (8)
C14*B*	0.5611 (3)	0.61013 (9)	0.7107 (3)	0.0568 (7)
C15*B*	0.5309 (2)	0.64999 (4)	0.7717 (2)	0.0556 (7)
C16*B*	0.4557 (2)	0.67657 (6)	0.67182 (14)	0.0618 (8)
C17*B*	0.4331 (2)	0.71547 (5)	0.7221 (2)	0.0676 (8)
C18*B*	0.4856 (2)	0.72779 (4)	0.8722 (2)	0.0648 (8)
C19*B*	0.5608 (2)	0.70121 (6)	0.97213 (15)	0.0664 (8)
C20*B*	0.5835 (2)	0.66231 (5)	0.9219 (2)	0.0589 (7)
C21*B*	1.0330 (4)	0.25160 (11)	0.6092 (4)	0.0752 (9)
C22*B*	1.0027 (6)	0.20003 (14)	0.4168 (6)	0.126 (2)
C23*B*	0.8648 (6)	0.18204 (15)	0.3511 (9)	0.163 (3)
C24*B*	0.8828 (10)	0.1416 (2)	0.2696 (9)	0.192 (3)‡
C25*B*†	0.869 (3)	0.1132 (5)	0.362 (2)	0.164 (7)‡
C25*B*′†	0.955 (3)	0.1126 (6)	0.372 (3)	0.166 (10)‡
C25*B*′′†	0.790 (2)	0.1113 (6)	0.272 (3)	0.152 (8)‡
C31*B*	0.5458 (5)	0.78313 (11)	1.0531 (4)	0.0840 (11)
C32*B*	0.5400 (5)	0.82605 (11)	1.0499 (4)	0.0936 (12)
C33*B*	0.6146 (6)	0.84719 (12)	1.1948 (5)	0.1053 (14)
C34*B*	0.6146 (7)	0.89051 (14)	1.1961 (6)	0.124 (2)
C35*B*	0.6864 (8)	0.91290 (15)	1.3372 (7)	0.152 (2)
C36*B*	0.6868 (9)	0.9553 (2)	1.3409 (8)	0.185 (3)
C37*B*	0.7637 (13)	0.9769 (2)	1.4818 (9)	0.261 (6)
C38*B*	0.7568 (13)	1.0195 (2)	1.4767 (12)	0.316 (7)

† Partially occupied site (see below). ‡ $U_{\rm iso}$.

Table 4. *Selected geometric parameters (Å, °) for the butyl compound*

O1*A*—C21*A*	1.335 (4)	O1*B*—C21*B*	1.329 (5)
O1*A*—C22*A*	1.444 (4)	O1*B*—C22*B*	1.431 (5)
O2*A*—C21*A*	1.205 (4)	O2*B*—C21*B*	1.198 (4)
O3*A*—C14*A*	1.365 (4)	O3*B*—C14*B*	1.361 (3)
O3*A*—C11*A*	1.383 (2)	O3*B*—C11*B*	1.383 (2)
O4*A*—C14*A*	1.204 (3)	O4*B*—C14*B*	1.200 (3)
O5*A*—C18*A*	1.367 (3)	O5*B*—C18*B*	1.356 (3)
O5*A*—C31*A*	1.434 (4)	O5*B*—C31*B*	1.432 (4)
N1*A*—C7*A*	1.261 (4)	N1*B*—C7*B*	1.275 (4)
N1*A*—C4*A*	1.423 (3)	N1*B*—C4*B*	1.419 (3)
C1*A*—C21*A*	1.478 (4)	C1*B*—C21*B*	1.469 (4)
C8*A*—C7*A*	1.463 (4)	C8*B*—C7*B*	1.451 (3)
C15*A*—C14*A*	1.463 (3)	C15*B*—C14*B*	1.464 (3)
C7*A*—N1*A*—C4*A*	118.8 (2)	C7*B*—N1*B*—C4*B*	117.3 (3)
N1*A*—C7*A*—C8*A*	123.5 (3)	N1*B*—C7*B*—C8*B*	123.8 (3)

For the ethyl compound (n = 2), all the non-H atoms were refined anisotropically. For the butyl compound (n = 4), the C25 atoms had remarkably large displacement parameters and considerably high peaks were found around them. Thus, these atoms were disordered in further refinements. Occupancies were fixed at 0.4 for atoms C25*A* and C25*B* and 0.3 for atoms C25*A*′, C25*A*″, C25*B*′ and C25*B*″. All the non-H atoms were refined anisotropically, except for C24*B* and the disordered C25 atoms. The butyl chains were refined with distance constraints due to the disordering. All the benzene rings were constrained to have regular hexagonal geometry, with C—C distances of 1.39 Å. The H atoms attached to atom C7 of the azomethine groups were found in the difference Fourier maps and refined isotropically, while all other H atoms (except for those attached to the C24 and C25 atoms of the butyl compound) were calculated geometrically (C—H distances were constrained to be 0.96 Å for primary H atoms, 0.97 Å for secondary and 0.93 Å for aromatic) and not refined.

For both compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992*a*); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1992*b*). Program(s) used to solve structures: *MULTAN*88 (Debaerdemaeker, Tate & Woolfson, 1985) for the ethyl compound; *MITHRIL*90 (Gilmore, 1984) for the butyl compound. For both compounds, program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *TEXSAN*; software used to prepare material for publication: *SHELXL*93 (Sheldrick, 1993).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1215). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Xanthurenic Acid Monohydrate

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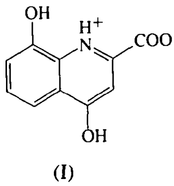
Abstract

In the structure of the title compound, 4,8-dihydroxy-2-quinolinecarboxylic acid monohydrate, $C_{10}H_7NO_4.H_2O$, the ionized carboxy group is located in the quinoline ring plane and the N atom of the ring is protonated so that it is positively charged, *i.e.* the compound may be named 4,8-dihydroxyquinolinium-2-carboxylate monohydrate. The crystal structure is stabilized by intermolecular O—H···O hydrogen bonds involving the water molecule.

Comment

Xanthurenic acid, (I), is a well known tryptophan metabolite which is induced from kynurenin as a result of vitamin B_6 deficiency. This metabolite occurs in the urine of humans, monkeys and rats when dietry vitamin B_6 is inadequate (Martin, Mayes & Rodwell, 1983). Xanthurenic acid has been reported to form a complex with insulin and damage pancreatic β cells (Kotake *et al.*, 1968; Murakami, 1968). Also, an increase in its formation may alter glucose metabolism (Seifert & Pewnim, 1992).



(I)

In order to elucidate the metabolic pathway of tryptophan metabolites, accurate structural and conformational information is indispensable. We have thus undertaken the structural analysis of the title compound.

The molecular structure is shown in Fig. 1, with the unit-cell packing in Fig. 2. The carboxy group is

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