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

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

	x	y	z	U_{eq}
Si1	0.08219 (11)	0.44582 (3)	0.4803 (3)	0.0676
O1	0.0684 (3)	0.41670 (7)	0.2955 (6)	0.0758
C1	−0.0336 (3)	0.32938 (9)	0.2162 (10)	0.0645
C2	−0.0299 (4)	0.36746 (10)	0.1921 (11)	0.0726
C3	0.0634 (4)	0.38161 (9)	0.3288 (9)	0.0661
C4	0.1742 (3)	0.36631 (10)	0.2569 (10)	0.0683
C5	0.1716 (3)	0.32842 (10)	0.2673 (8)	0.0580
C6	0.2516 (3)	0.31167 (10)	0.3669 (9)	0.0646
C7	0.2584 (3)	0.27486 (9)	0.3801 (9)	0.0634
C8	0.1790 (3)	0.25693 (9)	0.2247 (8)	0.0557
C9	0.0656 (3)	0.27450 (9)	0.2229 (7)	0.0506
C10	0.0736 (3)	0.31148 (10)	0.1489 (7)	0.0515
C11	−0.0210 (3)	0.25421 (10)	0.0955 (11)	0.0680
C12	−0.0308 (3)	0.21760 (10)	0.1655 (9)	0.0614
C13	0.0834 (3)	0.20003 (10)	0.1554 (7)	0.0529
C14	0.1629 (3)	0.22074 (9)	0.2952 (8)	0.0542
C15	0.2648 (3)	0.19915 (10)	0.3282 (10)	0.0680
C16	0.2181 (3)	0.16360 (10)	0.3314 (11)	0.0715
C17	0.0926 (3)	0.16619 (9)	0.2725 (9)	0.0589
C18	0.1225 (5)	0.19723 (12)	−0.0785 (10)	0.0743
C19	0.0929 (5)	0.31457 (12)	−0.0943 (9)	0.0736
C20	0.0493 (4)	0.13462 (10)	0.1605 (11)	0.0706
C21	−0.0767 (4)	0.13683 (11)	0.1116 (12)	0.0854
C22	0.0718 (5)	0.10262 (11)	0.3011 (13)	0.0874
C23	0.0060 (6)	0.10092 (13)	0.5023 (14)	0.1041
C24	−0.0533 (8)	0.07745 (17)	0.5850 (17)	0.1290
C25	0.0628 (7)	0.07117 (15)	0.1660 (19)	0.1186
C26	0.1675 (5)	0.43148 (17)	0.7151 (14)	0.1014
C27	0.1545 (7)	0.48062 (15)	0.3390 (14)	0.1073
C28	−0.0576 (5)	0.45955 (13)	0.5827 (11)	0.0860
C29	−0.1120 (7)	0.43049 (19)	0.708 (2)	0.1261
C30	−0.1311 (8)	0.4687 (3)	0.3865 (19)	0.1379
C31	−0.0458 (9)	0.4902 (2)	0.7324 (18)	0.1385

Table 2. *Selected geometric parameters* (Å, °)

Si1—O1	1.643 (4)	C10—C19	1.523 (7)
Si1—C26	1.867 (8)	C12—C11	1.539 (6)
Si1—C27	1.864 (7)	C13—C12	1.546 (5)
Si1—C28	1.878 (6)	C13—C17	1.545 (6)
O1—C3	1.427 (5)	C13—C18	1.522 (7)
C1—C2	1.539 (5)	C14—C13	1.533 (6)
C1—C10	1.533 (6)	C14—C15	1.515 (5)
C2—C3	1.514 (7)	C16—C15	1.536 (6)
C3—C4	1.533 (6)	C17—C16	1.556 (6)
C4—C5	1.526 (6)	C17—C20	1.537 (6)
C5—C6	1.326 (6)	C20—C21	1.547 (7)
C5—C10	1.545 (6)	C20—C22	1.576 (8)
C6—C7	1.485 (5)	C22—C23	1.473 (11)
C7—C8	1.534 (6)	C22—C25	1.519 (10)
C8—C9	1.535 (5)	C23—C24	1.288 (11)
C8—C14	1.531 (5)	C28—C29	1.548 (11)
C9—C11	1.539 (6)	C28—C30	1.543 (13)
C10—C9	1.559 (5)	C28—C31	1.546 (11)
O1—Si1—C26	112.0 (3)	C9—C11—C12	114.5 (4)
O1—Si1—C27	104.9 (3)	C13—C12—C11	111.0 (3)
O1—Si1—C28	110.6 (2)	C14—C13—C12	106.4 (3)
C26—Si1—C27	109.8 (3)	C14—C13—C17	99.8 (3)
C26—Si1—C28	108.7 (3)	C14—C13—C18	112.4 (4)
C27—Si1—C28	110.7 (3)	C12—C13—C17	116.6 (3)
Si1—O1—C3	127.5 (3)	C12—C13—C18	110.3 (4)
C2—C1—C10	114.6 (4)	C17—C13—C18	110.9 (4)
C1—C2—C3	110.0 (4)	C8—C14—C13	115.8 (4)
O1—C3—C2	108.8 (4)	C8—C14—C15	118.8 (3)
O1—C3—C4	108.6 (3)	C13—C14—C15	105.5 (3)
C2—C3—C4	109.3 (4)	C14—C15—C16	103.9 (3)
C3—C4—C5	111.8 (3)	C17—C16—C15	106.8 (3)
C4—C5—C10	115.9 (4)	C13—C17—C16	103.7 (3)
C4—C5—C6	120.8 (4)	C13—C17—C20	119.6 (4)
C10—C5—C6	123.3 (4)	C16—C17—C20	112.2 (3)
C5—C6—C7	124.9 (4)	C17—C20—C21	111.8 (4)
C6—C7—C8	113.6 (4)	C17—C20—C22	111.7 (5)
C7—C8—C9	109.9 (3)	C21—C20—C22	108.8 (4)
C7—C8—C14	110.4 (4)	C20—C22—C25	111.4 (6)
C9—C8—C14	109.1 (3)	C20—C22—C23	114.2 (4)
C10—C9—C8	112.8 (3)	C25—C22—C23	112.7 (5)
C10—C9—C11	113.4 (3)	C22—C23—C24	131.7 (7)
C8—C9—C11	111.1 (3)	Si1—C28—C29	108.9 (4)
C1—C10—C5	107.8 (3)	Si1—C28—C30	108.6 (5)
C1—C10—C9	108.5 (3)	Si1—C28—C31	110.7 (5)
C1—C10—C19	110.9 (4)	C29—C28—C30	109.4 (6)
C5—C10—C9	109.2 (3)	C29—C28—C31	109.9 (7)
C5—C10—C19	108.3 (4)	C30—C28—C31	109.4 (6)
C9—C10—C19	112.1 (4)		

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, torsion angles and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71544 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1074]

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2-(4-Pentanoyloxyphenyl)-6-pentanoyloxybenzoxazole

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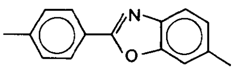
Abstract

In 2-[4-(pentanoyloxy)phenyl]benzoxazol-6-yl pentanoate, $C_{23}H_{25}NO_5$, the phenyl and benzoxazole rings are not strictly coplanar, the dihedral angle between the corresponding least-squares planes being 10.8 (6)°. A torsion angle of the *gauche* type is present in the aliphatic tail bonded to the benzo ring. Molecules of the title compound are packed in layers piled up along **a**. The lateral packing of the molecules shows face-to-face and face-to-edge contacts among the aromatic rings.

Comment

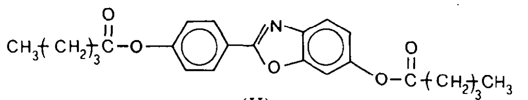
In the past few years, we have been investigating the phase behaviour of polymeric systems containing the group:



(I)

We have proved the mesogenic character of this group in a fully aromatic homopolyester (Caruso, Centore, Roviello & Sirigu, 1992) and in various classes of semiflexible polyesters (Caruso, Centore, Fresa, Roviello & Sirigu, 1992). The phase behaviour of semiflexible polyesters containing group (I) is particularly noteworthy. In fact, depending on the length of the flexible spacer, either the nematic phase or the smectic *A* phase, or both, may be observed. Furthermore, the observed layer periodicity of the smectic *A* phase is about half the length of the monomeric unit, suggesting a regularly intercalated packing of the chains in the smectic phase, which is quite unusual in the field of smectogenic polymers. We are interested in elaborating structural models for the crystal and, possibly, the smectic phase of these polymers; for this, the detailed knowledge of the structure of group (I) and of its modes of packing in crystalline low molar mass compounds that are chemically analogous to the polymers, seemed a prerequisite. The title compound (II) was chosen for a complete structural study, on the basis of its chemical similarity with polyesters of the formula —$[(CH_2)_n$—COO—*R*—OOC]—, whose phase behaviour has already been studied in our laboratory (Caruso, Centore, Fresa, Roviello & Sirigu, 1992).

The title compound was synthesized by esterification of 2-(4-hydroxyphenyl)-6-hydroxybenzoxazole, obtained as described previously (Caruso, Centore, Roviello & Sirigu, 1992), with pentanoyl chloride. It is not mesogenic and melts to an isotropic liquid at 379 K. Single crystals suitable for X-ray structure analysis were obtained by evaporation from chloroform/ethanol solutions.



(II)

Bond lengths and angles within the benzoxazole group are in fair agreement with literature data (Churchill, Wasserman & Young, 1980; Muir, Cox, Bernard & Muir, 1992). In particular, the pattern of the bond angles within the benzo ring shows some values significantly different from 120° [C13—C14—C15 = 126.1 (9), C14—C15—C16 = 113.5 (7)°]. At variance with the results found in a Pd complex of 2-*p*-tolylbenzoxazole (Churchill, Wasserman & Young, 1980), no distortion of the trigonal geometry around the C9 atom is observed. On the other hand, the trigonal geometry around the C12 atom is rather distorted in the title compound [N1—C12—C9 = 128.2 (9)°]; this may be due to the steric repulsion between N1 and the H atom bonded to C10 [N1···C10 = 2.929 (9) Å]. A similar, although quantitatively smaller, distortion is also found in the structure described by Muir, Cox, Bernard & Muir (1992), while in the structure reported by Churchill, Wasserman & Young (1980), the distortion of the trigonal geometry is reversed, probably as a result of the coordination geometry of the metal atom.

The benzoxazole group is planar to within 0.03 Å. The benzoxazole and the phenyl groups, however, are not coplanar, the dihedral angle between the corresponding least-squares planes being 10.8 (6)°. A slight puckering of group (I) is also present, as a consequence of the non-coplanarity of the C9 atom with the plane of the benzoxazole group [C13—N1—C12—C9 = 174.4 (5)°] and of atom C12 with the plane of the phenyl ring [C7—C8—C9—C12 = 174.7 (5)°]. The torsion angles around the C—O bonds contiguous to the phenyl and benzo rings [C5—O2—C6—C7 = 70.9 (7) and C19—O4—C16—C17 = −76.6 (7)°] are in the range expected on the basis of theoretical considerations (Hummel & Flory, 1980). The reliability of some bond lengths in the aliphatic tails of the molecule is poor (some C—C distances appear shorter than standard values); this is probably because of the high thermal motion affecting the terminal atoms of the chains. The C1–C5 chain has a *trans*-planar conformation, while a torsion angle of the *gauche* type is observed around bond C20—C21 [C19—C20—C21—C22 = 72.9 (9)°]. This should simply be a packing effect and results in a more linear molecule with an intrinsically non-linear rigid group (bonds C6—O2 and C16—O4 are, in fact, neither collinear nor parallel). The crystal packing (Fig. 2) may be described as an assembly of molecular layers piled up along **a**. Within each layer, the molecular axes are slightly tilted with respect to the normal to the layer surface, thus simulating a smectic *C*-type arrangement. In this respect, the absence of mesogenic behaviour in the title compound seems rather surprising. Along the **a** direction, layers of molecules are packed by feeble aliphatic–aliphatic van der Waals forces, without intercalation between the alkyl tails of the consecutive layers. This is consistent with the large thermal motion observed for the C atoms of the tails, which increases from the carbonyl C atom to the end of the chain. Within each layer, the packing is the result of dipolar interactions involving the carbonyl groups [O5···C5^{i} = 3.284 (9) Å; (i) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$] and of lateral interactions among the aromatic rings, which are of the face-to-face [C9···C17^{ii} = 3.359 (7), C12···C18^{ii} = 3.435 (8) Å; (ii) $1-x$, $-y$, $-z$] and face-to-edge types [C8···C14^{iii} = 3.544 (8) Å; (iii) $1-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$].

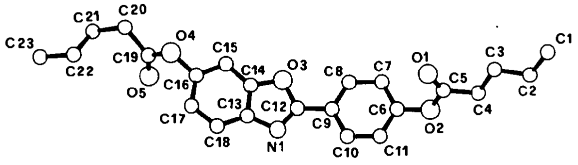


Fig. 1. Molecular drawing of the title compound showing the labelling of non-H atoms.

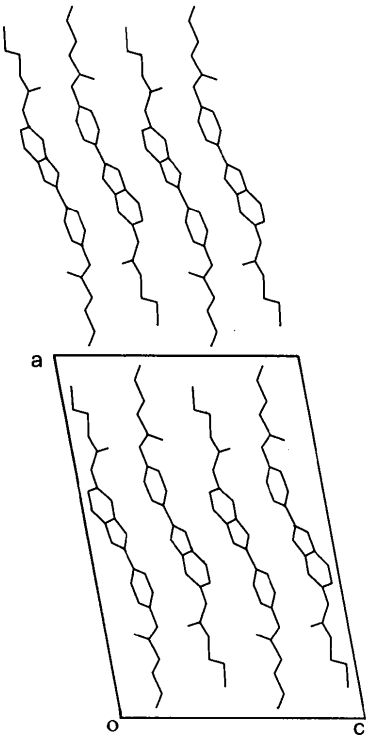


Fig. 2. Crystal packing viewed down [010].

Experimental

Crystal data

$C_{23}H_{25}NO_5$
M_r = 395.46
Monoclinic
$P2_1/c$
a = 23.920 (8) Å
b = 5.593 (2) Å
c = 16.028 (9) Å
β = 100.74 (4)°
V = 2107 (16) Å^3
Z = 4
D_x = 1.247 Mg m^{-3}

Mo $K\alpha$ (graphite monochromated) radiation
λ = 0.71069 Å
Cell parameters from 19 reflections
θ = 7.3- 12.3°
μ = 0.0820 mm^{-1}
T = 293 K
Thin plate
0.5 × 0.3 × 0.04 mm
Pale tan

Data collection

Enraf-Nonius CAD-4 diffractometer
ω/θ scans
Absorption correction: none
4630 measured reflections
4480 independent reflections
1008 observed reflections [$I > 3\sigma(I)$]

R_{int} = 0.021
$\theta_{\max}$ = 26.8°
$h = -30 \rightarrow 30$
$k = 0 \rightarrow 7$
$l = 0 \rightarrow 20$
2 standard reflections frequency: 60 min intensity variation: <5% (random)

Refinement

Refinement on F
R = 0.053
wR = 0.055
S = 1.555
1008 reflections
262 parameters
H-atom parameters not refined

$w = 4F_o^2/[\sigma^2(F_o^2) + (0.04F_o^2)^2]$
$(\Delta/\sigma)_{\max}$ = 0.01
$\Delta\rho_{\max}$ = 0.22 e Å^{-3}
$\Delta\rho_{\min}$ = −0.16 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$U_{\text{eq}} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
O1	0.2312 (2)	0.3111 (9)	0.1198 (3)	0.103 (2)
O2	0.2511 (2)	0.0218 (8)	0.2183 (2)	0.082 (2)
O3	0.5058 (2)	0.1016 (7)	0.1618 (2)	0.059 (1)
O4	0.6819 (2)	0.2158 (8)	0.0633 (2)	0.076 (2)
O5	0.7445 (2)	−0.003 (1)	0.1499 (3)	0.123 (2)
N1	0.4841 (2)	−0.2475 (9)	0.0946 (3)	0.067 (2)
C1	0.0285 (4)	0.585 (2)	0.1529 (6)	0.224 (6)
C2	0.0664 (3)	0.390 (2)	0.1862 (6)	0.173 (5)
C3	0.1245 (3)	0.402 (2)	0.1628 (5)	0.125 (4)
C4	0.1618 (3)	0.200 (1)	0.2024 (4)	0.107 (4)
C5	0.2172 (3)	0.190 (1)	0.1740 (4)	0.080 (3)
C6	0.3058 (3)	−0.001 (1)	0.2009 (4)	0.070 (3)
C7	0.3464 (3)	0.174 (1)	0.2259 (3)	0.069 (3)
C8	0.3997 (3)	0.143 (1)	0.2078 (3)	0.065 (3)
C9	0.4123 (3)	−0.060 (1)	0.1659 (3)	0.060 (2)
C10	0.3719 (3)	−0.236 (1)	0.1429 (3)	0.071 (3)
C11	0.3183 (3)	−0.206 (1)	0.1600 (3)	0.072 (3)
C12	0.4671 (3)	−0.082 (1)	0.1405 (3)	0.057 (2)
C13	0.5374 (3)	−0.173 (1)	0.0822 (3)	0.063 (2)
C14	0.5507 (2)	0.042 (1)	0.1227 (3)	0.056 (2)
C15	0.5997 (3)	0.173 (1)	0.1229 (3)	0.064 (3)
C16	0.6352 (3)	0.074 (1)	0.0748 (3)	0.059 (3)
C17	0.6250 (3)	−0.144 (1)	0.0339 (3)	0.067 (3)
C18	0.5756 (3)	−0.268 (1)	0.0361 (3)	0.066 (3)
C19	0.7350 (3)	0.161 (1)	0.1029 (4)	0.090 (3)
C20	0.7761 (3)	0.346 (2)	0.0793 (4)	0.119 (4)
C21	0.8388 (4)	0.276 (2)	0.0945 (5)	0.167 (5)
C22	0.8488 (4)	0.105 (2)	0.0343 (5)	0.192 (5)
C23	0.9136 (3)	0.054 (2)	0.0472 (6)	0.230 (8)

Table 2. *Selected geometric parameters* (Å, °)

O1—C5	1.196 (8)	C10—C11	1.370 (8)
O2—C5	1.354 (8)	C13—C14	1.376 (8)
O2—C6	1.393 (8)	C13—C18	1.382 (8)
O3—C12	1.382 (7)	C14—C15	1.384 (8)
O3—C14	1.381 (8)	C15—C16	1.366 (8)
O4—C16	1.408 (8)	C16—C17	1.386 (8)
O4—C19	1.346 (9)	C17—C18	1.375 (8)
O5—C19	1.182 (9)	C1—C2	1.45 (1)
N1—C12	1.296 (8)	C2—C3	1.51 (1)
N1—C13	1.387 (9)	C3—C4	1.505 (9)
C6—C7	1.387 (9)	C4—C5	1.48 (1)

C6—C11	1.379 (9)	C19—C20	1.52 (1)
C7—C8	1.371 (8)	C20—C21	1.52 (1)
C8—C9	1.378 (9)	C21—C22	1.41 (1)
C9—C10	1.382 (8)	C22—C23	1.55 (1)
C9—C12	1.447 (8)		
C5—O2—C6	116.9 (7)	N1—C13—C18	132.7 (8)
C12—O3—C14	104.5 (6)	C14—C13—C18	117.8 (9)
C16—O4—C19	120.8 (7)	O3—C14—C13	107.1 (9)
C12—N1—C13	105.2 (7)	O3—C14—C15	126.8 (8)
O1—C5—O2	123.5 (9)	C13—C14—C15	126.1 (9)
O1—C5—C4	126.2 (9)	C14—C15—C16	113.5 (7)
O2—C5—C4	110.3 (8)	O4—C16—C15	116.0 (7)
O2—C6—C7	120.7 (9)	O4—C16—C17	120.5 (9)
O2—C6—C11	117.8 (9)	C15—C16—C17	123.4 (9)
C7—C6—C11	121.4 (8)	C16—C17—C18	120.5 (8)
C6—C7—C8	118.6 (9)	C13—C18—C17	118.7 (7)
C7—C8—C9	120.2 (8)	O4—C19—O5	122 (1)
C8—C9—C10	120.7 (8)	O4—C19—C20	108.9 (9)
C8—C9—C12	119.9 (9)	O5—C19—C20	129 (1)
C10—C9—C12	119.3 (9)	C1—C2—C3	115. (1)
C9—C10—C11	119.6 (8)	C2—C3—C4	111.4 (9)
C6—C11—C10	119.4 (9)	C3—C4—C5	113.1 (8)
O3—C12—N1	113.8 (8)	C19—C20—C21	116.5 (9)
O3—C12—C9	117.9 (8)	C20—C21—C22	111. (1)
N1—C12—C9	128.2 (9)	C21—C22—C23	109. (1)
N1—C13—C14	109.4 (8)		
C6—O2—C5—O1	2.6 (9)	O2—C6—C11—C10	−179.2 (5)
C6—O2—C5—C4	−177.4 (5)	C7—C8—C9—C12	174.7 (5)
C5—O2—C6—C7	70.9 (7)	C12—C9—C10—C11	−174.2 (5)
C5—O2—C6—C11	−110.7 (6)	C8—C9—C12—O3	1.7 (8)
C19—O4—C16—C15	107.6 (6)	C8—C9—C12—N1	−173.4 (6)
C19—O4—C16—C17	−76.6 (7)	C10—C9—C12—O3	177.7 (5)
C16—O4—C19—O5	−1.8 (9)	C10—C9—C12—N1	2.6 (9)
C16—O4—C19—C20	−179.3 (5)	N1—C13—C18—C17	177.5 (6)
C13—N1—C12—C9	174.4 (5)	C14—C15—C16—O4	171.8 (5)
C1—C2—C3—C4	177.7 (7)	O4—C16—C17—C18	−171.5 (5)
C2—C3—C4—C5	174.1 (6)	O4—C19—C20—C21	−161.6 (5)
C3—C4—C5—O1	−7 (1)	O5—C19—C20—C21	21 (1)
C3—C4—C5—O2	173.5 (6)	C19—C20—C21—C22	72.9 (9)
O2—C6—C7—C8	179.6 (5)	C20—C21—C22—C23	175.4 (8)

H atoms were placed at calculated positions [riding model, C—H = 0.95 Å, $U(\mathrm{H}) = U_{\mathrm{eq}}(\mathrm{C})$]. A refinement against 3037 unique reflections having $I > 0$ gave $R = 0.211$, $wR = 0.091$ and $S = 1.305$ (weights as above), without significant changes in the final structural parameters. Program used to solve structure: *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Programs used to refine structure: *SDP* (Enraf-Nonius, 1985). Refinement was by full-matrix least-squares method. All programs were run on a Digital MicroVAX computer.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71446 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1045]

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1,3,5-Triethyl-1,3,5-triazine-2,4,6(1*H*,3*H*,5*H*)-trione, $C_9H_{15}N_3O_3$

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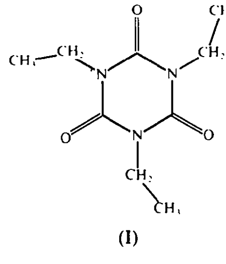
Abstract

The crystal structure of the title compound (triethylisocyanuric acid), which was obtained from the preparation of an ethyl isocyanate derivative of the anti-tumor compound CC-1065, has been determined by X-ray diffraction.

Comment

The title compound (I) was prepared accidentally during the synthesis of a derivative of the known anti-tumor compound CC-1065 (Chidester, Krüger, Mizsak, Duchamp & Martin, 1981). The attempted synthesis of an ethyl isocyanate derivative of CC-1065 yielded the crystalline compound triethylisocyanuric acid. The crystals appeared as clear yellow prisms from a reaction mixture of ethyl isocyanate and pyridine. The perhydrotriazine moiety is essentially planar with the terminal methyl groups lying



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