this is (+)-synclinal in the title compound. However, in classical 6,7-benzomorphans, conformations are restricted to the range between antiperiplanar and (−)-synclinal due to the unfavourable interactions between the C(3) and C(13) methylenes. Accordingly the C(13) methylene of the title compound occupies a volume that is normally free. It is conceivable that this volume exceeds the local boundaries of the receptor, thus leading to a global misalignment of the drug molecule. In this way, O(14) will be unable to form a hydrogen bond to the hypothetical common binding moiety in the receptor responsible for κ-opioid activity. The rather promising results from a fit between the title compound and ketazocine in free space thus make little sense in the physical environment of the receptor.

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Structure of 1-Pyrenebutanoic Acid

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Abstract. $C_{20}H_{16}O_2$, $M_r = 288.34$, monoclinic, $P2_1/a$, $a = 12.236$ (17), $b = 5.018$ (6), $c = 23.827$ (29) Å, $\beta = 92.26$ (11)°, $V = 1461.76$ (3.26) Å^3, $Z = 4$, $D_x = 1.310$ (2) g cm^{-3}, $\lambda(Cu K\alpha) = 1.54178$ Å, $\mu = 5.79$ cm^{-1}, $F(000) = 608.00$, room temperature. Data collected from twinned crystal; $h = \text{mod}(6)$ reflections overlapping. $R = 0.087$ for 936 reflections with $I > 3\sigma(I)$ and $h = \text{mod}(6)$ reflections excluded. Molecules related by the centre of inversion form hydrogen-bonded dimers, $H\cdots O = 1.64$ (1) Å, $O-H\cdots O = 149$ (1)°. The atoms participating in the hydrogen bond form an eight-membered ring which is almost flat

(except for the H atoms). The crystal has a Langmuir–Blodgett multilayer-type structure. The pyrene moieties pack in two neighbouring monolayers.

Introduction. A great deal of work has been carried out over the last few years attempting to elucidate the molecular order in Langmuir–Blodgett (LB) multilayers, for example Durfee, Storck, Willig & Frieling (1987), Bonnerot, Chollet, Frisby & Hoclet (1985), Swalen (1986). Yamazaki, Tamai & Yamazaki (1986) recently studied the excimer fluorescence of a mixed LB film that contained a fatty acid substituted pyrene moiety in order to quantify the extent of pyrene aggregation. To determine the details of the molecular packing in LB films it is very helpful to compare their spectra with those of a single crystal of the same chemical composition and known structure.

In this paper we report on the structure of a single crystal containing aromatic moieties, in this case pyrene, that are packed in a layered structure analogous to multilayers of LB films. A corresponding LB-type single crystal with anthracene as the aromatic moiety will be described elsewhere.

The pyrene moieties pack in two neighbouring monolayers, *i.e.* they form a bilayer, and are separated by butanoic acid chains which form hydrogen bonds between neighbouring layers. An unusual aspect of this structure is the packing of the pyrene moieties as monomeric units, as opposed to the face-to-face dimer arrangement observed for unsubstituted pyrene (Allmann, 1970).

This type of layered crystal should be extremely useful for studying the special properties of excited states, in ordered two-dimensional systems, *e.g.* lifetime and transport of excitation energy (Agranovich, 1984; Turlet, Kottis & Philpott, 1983), and of charge carriers. A comparison of the optical properties and dynamic behaviour of excited states in the present crystal with the excimers in the dimeric pyrene crystal will be particularly interesting.

Experimental. Slightly yellow crystals were obtained by slow evaporation of a THF/ethyl acetate/ethanol solution. The crystal dimensions were approximately $0.5 \times 0.4 \times 0.2$ mm. Cell parameters and intensity data measured on a Stoe diffractometer. The orientation matrix was determined from 15 carefully centred reflections with $\theta_{max} = 20°$, using Cu $K\alpha$, range of h, k, and l $-13 \rightarrow 13$, $0 \rightarrow 5$, $0 \rightarrow 26$, respectively, total of 1758 independent reflections measured [$h = $ mod(6) excluded] to $(\sin\theta)/\lambda = 0.56$ Å^{-1}, data not corrected for absorption (controls: orientation matrix and intensity $\overline{2}00$, max. change 2·3%); 936 reflections with $I > 3\sigma(I)$ used in refinement, $R_{int} = 0.017$. The structure was solved by direct methods using the *EEES* procedure of program *SHELX*76 (Sheldrick, 1976); the first E map revealed positions of all non-H atoms, least-squares

Table 1. *Final positional* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($\times 10^3$) *with e.s.d.'s in parentheses*

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

	x	y	z	$U_{eq}(\text{Å}^2)$
C1	5721 (8)	4337 (25)	3086 (5)	60 (12)
C2	4923 (10)	6240 (31)	3143 (6)	84 (15)
C3	4495 (10)	7699 (25)	2689 (6)	82 (15)
C4	4447 (9)	8703 (26)	1653 (6)	87 (13)
C5	4811 (10)	8235 (27)	1151 (6)	89 (15)
C6	6025 (11)	5619 (27)	528 (5)	60 (14)
C7	6794 (11)	3704 (29)	457 (5)	81 (15)
C8	7210 (10)	2299 (25)	914 (6)	74 (14)
C9	7246 (8)	1282 (19)	1919 (5)	61 (10)
C10	6906 (8)	1781 (22)	2446 (5)	60 (11)
C11	6093 (6)	3811 (21)	2541 (5)	45 (10)
C12	4857 (9)	7260 (22)	2149 (6)	68 (12)
C13	5628 (9)	6258 (22)	1055 (5)	55 (11)
C14	6843 (8)	2737 (20)	1454 (5)	57 (11)
C15	6050 (8)	4732 (19)	1531 (4)	43 (9)
C16	5665 (8)	5254 (19)	2073 (5)	53 (10)
C101	6160 (10)	2904 (25)	3605 (5)	55 (14)
C102	7264 (8)	4050 (25)	3817 (4)	54 (12)
C103	7665 (10)	2529 (21)	4340 (5)	72 (13)
C104	8715 (9)	3605 (23)	4613 (5)	64 (12)
O1	9131 (6)	5713 (15)	4465 (3)	62 (8)
O2	9090 (6)	2171 (15)	5034 (3)	79 (9)

Table 2. *Interatomic distances* (Å) (*e.s.d.'s in parentheses*)

C1	C2	1·376 (16)	C10	C11	1·448 (14)
C1	C11	1·418 (14)	C11	C16	1·413 (13)
C1	C101	1·510 (16)	C12	C16	1·428 (14)
C2	C3	1·392 (18)	C13	C15	1·446 (13)
C2	C12	1·396 (17)	C14	C15	1·410 (13)
C4	C5	1·314 (18)	C15	C16	1·416 (13)
C4	C12	1·457 (17)	C101	C102	1·536 (13)
C5	C13	1·434 (17)	C102	C103	1·526 (14)
C6	C7	1·360 (16)	C103	C104	1·516 (14)
C6	C13	1·400 (15)	C104	O1	1·231 (12)
C7	C8	1·376 (17)	C104	O2	1·304 (13)
C8	C14	1·397 (15)	O2	H21	1·12 (1)
C9	C10	1·362 (14)	O2	O1*	2·662 (11)
C9	C14	1·402 (14)	H21	O1*	1·64 (1)

* Symmetry-related atoms.

Table 3. *Bond angles* (°) (*e.s.d.'s in parentheses*)

C2	C1	C11	118·4 (1·2)	C6	C13	C15	117·1 (1·1)
C2	C1	C101	118·6 (1·2)	C8	C14	C9	122·0 (1·0)
C11	C1	C101	123·0 (1·1)	C8	C14	C15	118·7 (1·1)
C1	C2	C3	122·3 (1·3)	C9	C14	C15	119·3 (1·0)
C2	C3	C12	120·8 (1·2)	C13	C15	C14	119·9 (1·1)
C5	C4	C12	122·1 (1·3)	C13	C15	C16	119·7 (1·0)
C4	C5	C13	122·2 (1·3)	C14	C15	C16	120·3 (9)
C7	C6	C13	122·7 (1·1)	C11	C16	C12	119·9 (1·1)
C6	C7	C8	119·9 (1·2)	C11	C16	C15	119·9 (9)
C7	C8	C14	121·7 (1·2)	C12	C16	C15	120·2 (1·0)
C10	C9	C14	121·5 (1·0)	C1	C101	C102	111·8 (9)
C9	C10	C11	120·7 (1·0)	C101	C102	C103	109·4 (1·0)
C1	C11	C10	121·4 (1·0)	C102	C103	C104	114·3 (9)
C1	C11	C16	120·4 (1·0)	C103	C104	O1	122·4 (1·1)
C10	C11	C16	118·2 (1·0)	C103	C104	O2	113·5 (1·0)
C3	C12	C4	124·0 (1·3)	O1	C104	O2	123·9 (1·1)
C3	C12	C16	118·3 (1·2)	C104	O2	H21	117 (1)
C4	C12	C16	117·7 (1·2)	C104	O2	O1*	112·4 (7)
C5	C13	C6	124·8 (1·2)	O2	O1*	C104*	123·7 (7)
C5	C13	C15	118·1 (1·1)	O2	H21	O1*	149 (1)

* Symmetry-related atoms.

Table 4. *Selected torsion angles* (°) *(e.s.d.'s in parentheses)*

H21*	O1	C104	O2	−9·2(8)	O2*	O1	C104	O2	0·0(6·9)
O1	C104	O2	H21	19·3(1·7)	O1	C104	O2	O1*	0·0(3·4)
C104	O2	H21	O1*	−8·7(7)	C104	O2	O1*	C104*	0·0(2·8)
O2	H21	O1*	C104*	10·1(9)					

* Symmetry-related atoms.

Fig. 1. Atom numbering.

Fig. 2. Stereoview showing the molecular packing.

refinement, isotropic, and then anisotropic temperature factors. H atoms located on difference Fourier map and, because of small number of reflections, included in the refinement, based on F, as a rigid body only. Unit weights. Refinement converged with $R = 0.087$ and $S = 1.78$; in the last cycle of refinement $(\Delta/\sigma)_{max}$ was 0·01 for all refined parameters; max., min. height on final difference Fourier map = 0·26, -0.25 e Å^{-3}. Scattering factors from *SHELX*76. Atomic coordinates are given in Table 1, interatomic distances in Table 2, angles in Table 3, and selected torsion angles in Table 4. Atom numbering is shown in Fig. 1, molecular packing in Fig. 2.*

Discussion. The molecule of the title compound consists of two parts: the pyrene and the saturated fatty acid which is attached to the pyrene (see Fig. 1). The pyrene is planar. The distances calculated from the best plane taken through the C atoms are not greater than 0·02 (2) Å for C atoms and 0·30 (14) Å for H atoms. The bond distances and angles are similar to those found in unsubstituted pyrene (Allmann, 1970).

There are four molecules in the unit cell. Two molecules are linked together by a hydrogen bond. The H···O distance = 1·64 Å and O—H···O angle = 149·1 (1)°. The atoms participating in the hydrogen bond (C104, O1, O2, H21 and symmetry-related atoms) form an eight-membered ring which is almost flat except for the H atoms. The distances from the best plane taken through the non-H atoms are not greater than 0·003 (13) Å for non-H atoms and 0·33 (13) Å for H atoms. (For planarity see also the torsion angles included in Table 4.)

Unsubstituted pyrene forms face-to-face dimers in the crystal, separated by 3·47 Å, with dimer units packed in a staggered array (Allmann, 1970). Fig. 2, however, shows the monomer arrangement of the pyrene groups. The packing of the title compound is thus more closely related to that observed for naphthalene, anthracene, *etc.* (Cruickshank, 1957; Mason, 1964) than to pyrene. The closest non-H intermolecular contact is 3·54 (2) Å and is between C6

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51629 (49 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
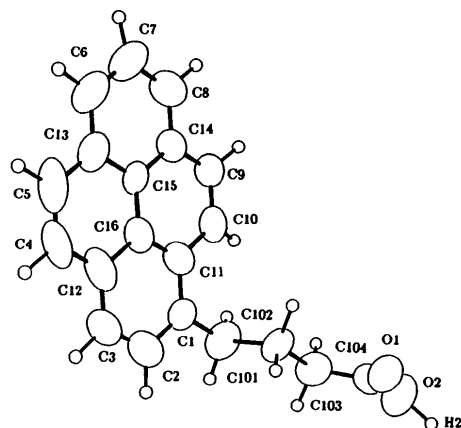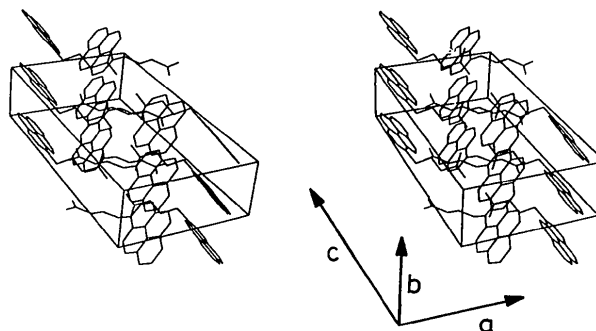
atoms related by an inversion centre. The edge-to-edge distance separating pyrene bilayers across the butanoic acid chains (determined by planes parallel to *ab* planes and through C1 atoms) is 9·11 (1) Å.

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