Table 3. *Hydrogen-bond lengths and angles in the crystal structure of methyl carbamate*

Distances in Å, angles in °. H···O$^r$ normalized values with N—H bond lengths extended to 1·03 Å, and C—H bond lengths extended to 1·09 Å.

| Donor Acceptor | H···O | H···O$^r$ | N—H···O | Symmetry of acceptor molecule |
|---|---|---|---|---|
| NH(4)···O=C | 2·14 (2) | 1·95 | 155 (2) | $1+x, y, z$ |
| NH(5)···O=C | 2·11 (2) | 1·93 | 173 (2) | $-x, 2-y, 1-z$ |
| C(1)H(3)···O(1) | 2·60 (2) | 2·51 | 158 (2) | $-1-x, -y, -z$ |

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# Structure of 9-Acetoxy-10-(4-acetoxyphenyl)anthracene at 95 K*

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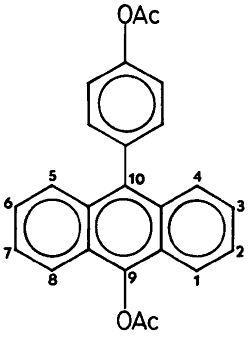
**Abstract.** $C_{24}H_{18}O_4$, $M_r = 370{\cdot}4$, monoclinic, $P2_1/n$, $a = 11{\cdot}021$ (2), $b = 18{\cdot}518$ (4), $c = 9{\cdot}125$ (2) Å, $\beta = 102{\cdot}99$ (2)°, $V = 1814{\cdot}6$ (7) Å$^3$, $Z = 4$, $D_m = 1{\cdot}31$ (2), $D_x = 1{\cdot}356$ (1) Mg m$^{-3}$, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}53$ cm$^{-1}$, $F(000) = 776$, $T = 95$ K. Final $R = 0{\cdot}049$ for 4727 unique observed reflections. The anthracene and phenyl rings exhibit deformations consistent with the σ-electron characteristics of their substituents. The dihedral angle between the phenyl and the anthracene ring system [84·4 (1)°] is sensitive to phenyl ring substitution and may have a bearing on the fluorescent properties of compounds in this series.

**Introduction.** The structure of the title compound (I) was determined as a part of investigations of 9-acetoxy-10-arylanthracenes, which exhibit marked fluorescence in organic solvents (Heldt, Heldt & Gronowska, 1975; Gronowska, Aleksandrzak & Heldt, 1981) and are used as active media for dye lasers (Heldt, 1983). Knowledge of the three-dimensional structures of these compounds could contribute to an improved understanding of the molecular basis for the influence of phenyl ring substitution on laser activity (Heldt, 1983).



(I)

**Experimental.** Light-brown rhombic crystals grown from ethanol solution by slow evaporation, m.p. 470·0–471·0 K; space group and approximate cell dimensions determined from Weissenberg and oscillation photographs; density measured by flotation in aqueous KI solution; crystal size: 0·20 × 0·28 × 0·60 mm; Syntex *P*3 diffractometer with a low-

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* IUPAC name: 10-(4-acetoxyphenyl)-9-anthryl acetate.

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temperature device (95 K), $\theta$–$2\theta$ scan mode, lattice parameters determined using 25 reflections in range $25 < 2\theta < 30°$; no absorption correction; max. $(\sin\theta)/\lambda = 0{\cdot}7053$ Å$^{-1}$, $-15 \leq h \leq 15$, $0 \leq k \leq 26$, $0 \leq l \leq 12$; four intensity standards [600: 255 (12); 0,14,0: 1103 (39); 004: 775 (38); 47$\bar{4}$: 2400 (48)]; 5006 unique reflections measured, 279 unobserved reflections ($I < \sigma_I$). Solution by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), only one set of phases generated and accepted as true solution. Full-matrix least-squares refinement using $F$ magnitudes with *SHELX*76 (Sheldrick, 1976); all H atoms located on difference maps, two sets of methyl H atoms found for acetoxy group bonded to phenyl ring, site-occupancy factor (equal for all three H atoms of one set with complement to 1·0 for second set) was varied to achieve similar values of isotropic displacement parameters for both sets, single displacement parameter for three H atoms of each set, final s.o.f. 0·47 (4) and 0·53 (4) for these two sets. Anisotropic displacement parameters for non-H atoms and isotropic for all H atoms refined. Final $R = 0{\cdot}049$, $wR = 0{\cdot}046$ and $S = 0{\cdot}82$ for 4727 observed reflections, unit weights, $(\Delta/\sigma)_{\max} = 0{\cdot}22$ for disordered methyl hydrogens and 0·03 for other atoms; $(\Delta\rho)_{\max} = 0{\cdot}40$ and $(\Delta\rho)_{\min} = -0{\cdot}24$ e Å$^{-3}$ in final difference Fourier map, all the highest peaks located approximately in the middle of bonds; one strong reflection, 210, omitted for possible extinction effects; atomic scattering factors those incorporated in *SHELX*76 (Sheldrick, 1976). Other computer programs used: *CRYPOZ* system (Jaskólski, 1982) and the data processing program of Blessing (1986).

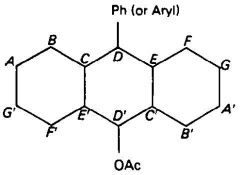
**Discussion.** The final atomic parameters are given in Table 1.* The bond lengths, bond angles and numbering scheme are shown in Fig. 1. Average geometric parameters for the anthracene moiety of the title structure (I), 9-acetoxy-10-(2,4-diacetoxyphenyl)-anthracene (II) (Roszak & Borowiak, 1986), 'unsubstituted' anthracenes (III) and '9,10-$sp^2$-disubstituted' anthracenes (IV) from Harlow, Loghry, Williams & Simonsen (1975) are compared in Table 2. When the bond lengths of (I) are compared with those of (II) and (IV) most of the differences are within three standard deviations and are associated with the foreshortening of bonds in (II) and (IV) due to thermal effects. The most significant structural differences in (I) compared with (III) are in the shortening of the *C'D'*

Table 1. *Final fractional coordinates and equivalent isotropic displacement parameters* (Å$^2$)

$U_{\rm eq} = (U_{11}U_{22}U_{33})^{1/3}$.

| | $x$ | $y$ | $z$ | $U_{\rm eq}$ |
|---|---|---|---|---|
| C(1) | 0·6061 (1) | 0·08952 (7) | 0·4690 (2) | 0·0173 (4) |
| C(2) | 0·5640 (1) | 0·08141 (8) | 0·3178 (2) | 0·0203 (4) |
| C(3) | 0·5402 (1) | 0·14253 (8) | 0·2215 (2) | 0·0200 (4) |
| C(4) | 0·5611 (1) | 0·21061 (8) | 0·2793 (1) | 0·0171 (4) |
| C(5) | 0·7047 (1) | 0·36955 (8) | 0·7223 (2) | 0·0179 (4) |
| C(6) | 0·7560 (1) | 0·37683 (8) | 0·8724 (2) | 0·0211 (4) |
| C(7) | 0·7848 (1) | 0·31518 (8) | 0·9662 (2) | 0·0200 (4) |
| C(8) | 0·7577 (1) | 0·24771 (8) | 0·9085 (1) | 0·0172 (4) |
| C(9) | 0·6757 (1) | 0·17027 (7) | 0·6868 (1) | 0·0146 (3) |
| C(10) | 0·6331 (1) | 0·29168 (7) | 0·4986 (1) | 0·0138 (3) |
| C(11) | 0·6791 (1) | 0·30002 (7) | 0·6548 (1) | 0·0151 (3) |
| C(12) | 0·7034 (1) | 0·23827 (7) | 0·7515 (1) | 0·0147 (3) |
| C(13) | 0·6294 (1) | 0·15988 (7) | 0·5333 (1) | 0·0143 (3) |
| C(14) | 0·6081 (1) | 0·22217 (7) | 0·4371 (1) | 0·0141 (3) |
| C(15) | 0·6151 (1) | 0·35652 (7) | 0·3986 (1) | 0·0142 (3) |
| C(16) | 0·7084 (1) | 0·37721 (7) | 0·3261 (1) | 0·0175 (4) |
| C(17) | 0·6947 (1) | 0·43885 (8) | 0·2360 (1) | 0·0178 (4) |
| C(18) | 0·5873 (1) | 0·47917 (7) | 0·2186 (1) | 0·0153 (4) |
| C(19) | 0·4923 (1) | 0·45899 (8) | 0·2862 (2) | 0·0210 (4) |
| C(20) | 0·5069 (1) | 0·39779 (8) | 0·3770 (2) | 0·0202 (4) |
| C(21) | 0·6117 (1) | 0·08497 (7) | 0·8458 (1) | 0·0164 (4) |
| C(22) | 0·6594 (2) | 0·02330 (8) | 0·9481 (2) | 0·0213 (4) |
| C(23) | 0·5936 (1) | 0·60588 (7) | 0·1911 (2) | 0·0176 (4) |
| C(24) | 0·5667 (2) | 0·66520 (8) | 0·0765 (2) | 0·0220 (4) |
| O(1) | 0·70335 (9) | 0·10945 (5) | 0·7800 (1) | 0·0162 (3) |
| O(2) | 0·5093 (1) | 0·11031 (6) | 0·8214 (1) | 0·0266 (3) |
| O(3) | 0·5723 (1) | 0·54008 (5) | 0·1240 (1) | 0·0179 (3) |
| O(4) | 0·6288 (1) | 0·61369 (6) | 0·3243 (1) | 0·0221 (3) |

Table 2. *A comparison of bond lengths* (Å) *and bond angles* (°) *for the anthracene moiety of the title compound* (I) *with those of* 9-*acetoxy*-10-(2,4-*diacetoxyphenyl*)*anthracene* (II) (*Roszak & Borowiak,* 1986) *and with those from Harlow et al.* (1975) *for 'average unsubstituted'* (III) *and 'average* 9,10-$sp^2$-*disubstituted'* (IV) *anthracene*



| | (I)* | (II)* | (III)† | (IV)† |
|---|---|---|---|---|
| *AB* | 1·364 (1) | 1·352 (4) | 1·367 | 1·354 |
| *BC* | 1·431 (2) | 1·429 (2) | 1·433 | 1·432 |
| *CD* | 1·408 (2) | 1·406 (3) | 1·399 | 1·402 |
| *C'D'* | 1·394 (2) | 1·386 (1) | | |
| *AG'* | 1·420 (1) | 1·413 (2) | 1·419 | 1·413 |
| *CE'* | 1·435 (2) | 1·435 (1) | 1·433 | 1·438 |
| *G'AB* | 120·6 (2) | 120·5 (2) | 120·8 | 120·4 |
| *ABC* | 121·2 (1) | 121·3 (2) | 120·3 | 121·4 |
| *A'B'C'* | 120·4 (1) | | | |
| *BCE'* | 117·7 (1) | 117·6 (1) | 118·9 | 118·0 |
| *B'C'E* | 119·7 (1) | 118·9 (2) | | |
| *BCD* | 122·1 (1) | 122·8 (3) | 121·7 | 122·2 |
| *E'CD* | 120·4 (4) | 120·0 (5) | 119·5 | 119·8 |
| *EC'D'* | 118·1 (3) | 118·1 (4) | | |
| *CDE* | 119·8 (1) | 120·2 (2) | 121·0 | 120·5 |
| *C'D'E'* | 123·1 (1) | 123·6 (2) | | |

* *mm*2 symmetry assumed except for the parameters *AB*, *BC*, *G'AB* and *BCD* for which the distribution of values seems to be random and *mmm* symmetry assumed.

† *mmm* symmetry assumed.

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* Lists of structure factors, anisotropic displacement parameters, H-atom parameters, least-squares-plane data, intermolecular distances and Fig. 2 illustrating the non-planarity of the anthracene ring system have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43312 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

and *D'E'* bonds and opening of the *C'D'E'* angle and in the lengthening of the *CD* and *DE* bonds and closing of the *CDE* angle associated with the substitution of the acetoxy and 4-acetoxyphenyl groups, respectively. This marked difference in geometries about the carbon atoms in positions 9 and 10 of the anthracene is due to the $\sigma$-electron-withdrawing character of the OAc group (Roszak & Borowiak, 1986) and the $\sigma$-electron-releasing character of the phenyl group (Domenicano & Murray-Rust, 1979).

The anthracene ring system is not strictly planar, as in the case of other non-centrosymmetric anthracene derivatives, *e.g.* 9-methylanthracene (Cox & Sim, 1979) and its derivatives (Glusker & Zacharias, 1972; Stallings, Monti & Glusker, 1982) and 9-{[(2-chloroethyl)thio]methyl}anthracene (Lewis, Carrell, Glusker & Sparks, 1976). The ring is slightly buckled (Fig. 2)* and the r.m.s. deviation from the least-squares plane through the 14 C atoms of the anthracene is 0·037 Å.

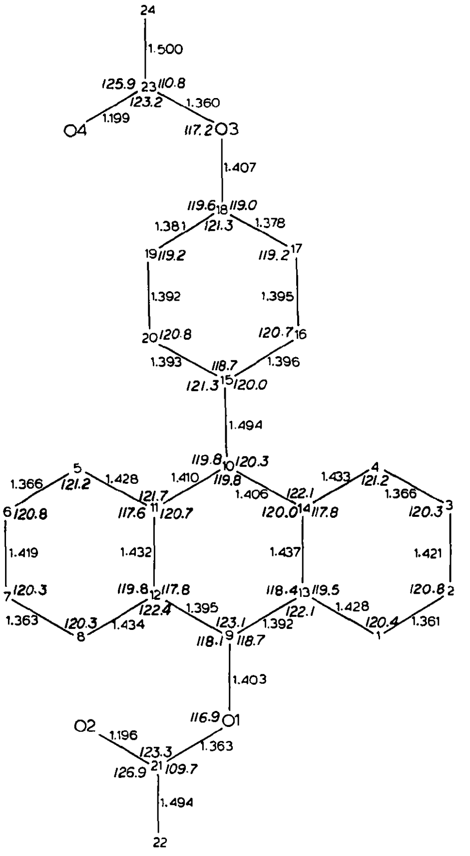

Fig. 1. Bond lengths (Å) and bond angles (°) with the numbering scheme; e.s.d.'s are 0·001–0·002 Å and 0·1°. The C—H bond distances range from 0·91 to 1·04 Å with an average of 0·97 (3) Å.

None of the individual rings are strictly planar. The planes of the outer rings form a dihedral angle of 3·6 (1)° and atoms O(1) and C(15) are displaced from the least-squares plane of the central ring by 0·063 (1) and 0·056 (1) Å. Similar deformations were observed in (II) and in 9-acetoxy-10-(2-acetoxy-5-bromophenyl)-anthracene (V) (Roszak & Skrzat, 1985).

The influence of the 9-acetoxy-10-anthryl and acetoxy groups on the internal angles of the phenyl ring is consistent with their $\sigma$-electron character and the general theory of such substitution effects (Domenicano, Vaciago & Coulson, 1975). The magnitudes of the internal angles at C(15) and C(18) are in very good agreement with values of 118·6 and 121·3° calculated using the angular substituent parameters evaluated on the basis of five analogous structures (Roszak, 1986). Very similar values of 118·9 (2) and 121·5 (2)° for analogous internal angles in the phenyl ring have been found in the structure of 10-(4-acetoxyphenyl)-10-hydroxy-9(10*H*)-anthracenone (Skrzat & Roszak, 1986).

The phenyl ring forms a dihedral angle of 84·4 (1)° with the anthracene ring system. In structures in which there are acetoxy groups substituted on the phenyl ring in the *ortho* position relative to the anthracene

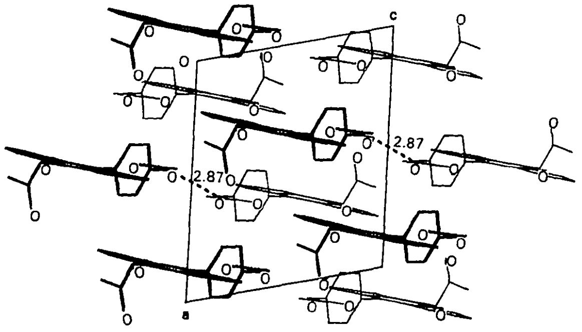

Fig. 3. View of the crystal structure along **b**. Distance in Å.

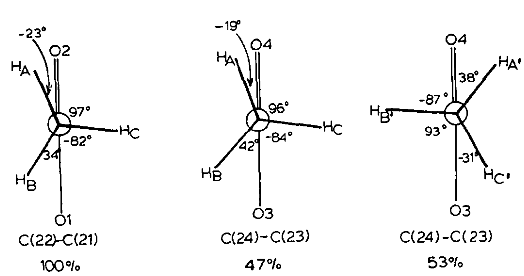

Fig. 4. Newman projections for methyl hydrogens; site-occupancy factor is marked below. E.s.d.'s are 1·5 and 3° for C(22) and C(24) hydrogens, respectively.

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* Fig. 2 has been deposited. See deposition footnote.

substituent dihedral angles of 68·2 (II), 69·9 (V*a*) and 64·8° (V*b*) have been observed. Favourable non-bonding interactions between the *ortho* substituent and the anthracene must be stabilizing this conformation (Heldt, 1983), although in (V) some intermolecular interactions also play an important role.

All bond lengths, bond angles and torsion angles in the acetoxy groups are within one standard deviation of the average values calculated from a sample of 102 acetoxy groups bonded to aromatic six-membered carbon rings (Roszak, 1986).

The acetoxy group bonded to the anthracene is almost perpendicular to it [dihedral angle 89·2 (1)°]. A similar conformation is observed in (V) (89·7 and 86·3° for the two molecules in the asymmetric unit). The OAc bonded to the phenyl ring forms a dihedral angle of 81·3 (1)° with it, resembling the conformation observed for (II).

Fig. 3 illustrates the packing of the title compound in the unit cell. The arrangement of the methyl H atoms in relation to the plane of the OAc group is shown in Fig. 4. It resembles the conformation found for methyl H atoms in the rhombohedral form of acetamide [neutron study at 23 K: Jeffrey, Ruble, McMullan, DeFrees, Binkley & Pople (1980)].

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*Acta Cryst.* (1987). **C43**, 254–256

# Structure of 1-Nitro-7*H*-benzo[*h,i*]chrysen-7-one

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**Abstract.** $C_{21}H_{11}NO_3$, $M_r = 325{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 16{\cdot}407$ (4), $b = 11{\cdot}909$ (4), $c = 7{\cdot}407$ (2) Å, $V = 1447{\cdot}3$ (7) Å$^3$, $Z = 4$, $D_m = 1{\cdot}46$, $D_x = 1{\cdot}493$ Mg m$^{-3}$, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 10{\cdot}9$ mm$^{-1}$, $F(000) = 672$, $T = 298$ K. Final $R = 0{\cdot}059$ for 1220 independent reflections. The molecule is greatly distorted from a planar structure owing to the overcrowding around the nitro and carbonyl groups. The mean planes of the two naphthalene moieties make an angle of 25·7 (2)°. The $NO_2$ group rotates around the C—N bond from a coplanar conformation. The torsion angle O—N—C—C is 45·7 (3)°.

**Introduction.** Nitration of ketones of condensed polycyclic aromatic compounds gives various nitro derivatives. It is often difficult, however, to assign the

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