

butadiène-1,3 (1,48 Å) et le propenal (1,45 Å); la molécule présente donc un caractère de simple liaison plus accentué que dans ces dernières molécules, phénomène qui selon Eriks *et al.* (1983) serait attribuable à la contribution de la forme résonante $O^- - C^+(R) - C^+(R) - O^-$ dans la structure électronique des dérivés dicarbonylés.

La quasi invariance de la distance entre les deux carbonyles lorsqu'on passe de la dicétone à son dérivé dichloré (respectivement 1,540 et 1,527 Å) donne à penser que la substitution des atomes de chlore en α du carbonyle ne modifie pas la conjugaison entre les deux groupes $C=O$ de façon appréciable.

Comme pour la butanedione-2,3 mais de façon moins marquée, la liaison $C(1) - C(2)$ est plus courte que $C(2) - C(2')$; l'écart est de 0,025 Å pour le dérivé dihalogéné et de 0,065 Å pour la dicétone non substituée.

En conclusion, la structure de la dichloro-1,4 butanedione-2,3 à l'état solide se caractérise par une disposition antipériplanaire de ses deux groupes carbonyle et une orientation synpériplanaire des groupes $C=O$ et $C-Cl$ adjacents. Nous montrerons dans un prochain mémoire que les conformères centrosymétriques de ce composé ont un moment dipolaire incompatible avec leur structure tout comme la dicétone non substituée.

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Références

- BLOOM, G. I. M. & SUTTON, L. E. (1941). *J. Chem. Soc.* pp. 727-742.
 DANIELSON, D. D. & HEDBERG, K. (1979). *J. Am. Chem. Soc.* **101**(14), 3730-3734.
 DURIG, J. R., HANNUM, S. E. & BROWN, S. C. (1971). *J. Phys. Chem.* **75**(13), 1946-1956.
 ERIKS, K., HAYDEN, T. D., HSI YANG, S. & CHAN, I. Y. (1983). *J. Am. Chem. Soc.* **105**(12), 3940-3947.
 HAGEN, K. & HELDBERG, K. (1973). *J. Am. Chem. Soc.* **95**(25), 8266-8269.
 JOHNSON, C. K. (1965). Rapport ORNL-3794. Oak Ridge National Laboratory, Tennessee.
 MAAS, K. (1898). *Chem. Zentralbl.* **1**, 24-25.
 MAURY, C., LERE-PORTE, J. P. & PETRISSANS, J. (1986). A paraître.
 NOACK, K. & JONES, R. N. (1960). *Z. Elektrochem.* **64**(5), 707-713.
 SHELDRIK, G. M. (1976). *SHELX76*. Programme pour la détermination des structures cristallines. Univ. de Cambridge, Angleterre.
 SØRENSEN, A. M. (1974). *Acta Cryst.* **B30**, 1366-1368.
 ZAHN, C. T. (1932). *Phys. Rev.* **40**, 291-298.
 ZALKIN, A. (1968). Berkeley, Californie. Travaux non publiés.

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Structure of 9-Acetoxy-10-(2,4-diacetoxyphenyl)anthracene*

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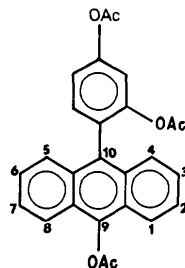
Abstract. $C_{26}H_{20}O_6$, $M_r = 428.45$, triclinic, $P\bar{1}$, $a = 10.513$ (2), $b = 14.325$ (2), $c = 8.785$ (1) Å, $\alpha = 105.16$ (1), $\beta = 120.90$ (1), $\gamma = 93.47$ (1)°, $V = 1065.0$ (3) Å³, $Z = 2$, $D_m = 1.33$ (1), $D_x = 1.336$ Mg m⁻³, m.p. = 457.2-458.7 K, $\lambda(Cu K\alpha) = 1.54178$ Å, $\mu = 0.70$ mm⁻¹, $F(000) = 448$, room temperature, $R = 0.042$ for 2465 observed reflections. The influence of substituents on the internal angles of the benzene ring was determined by using the analysis proposed by Domenicano & Murray-Rust [*Tetrahedron Lett.* (1979), pp. 2283-2286]. The geom-

etry of the 9,10-disubstituted anthracene ring system has been analysed and compared with that of unsubstituted anthracene.

Introduction. 9-Acetoxy-10-arylanthracenes, which all exhibit a marked fluorescence in organic solvents (Gronowska, Aleksandrak & Heldt, 1981), have been investigated as active media in dye lasers (Heldt, 1983). It was found (Heldt, 1983) that of the above compounds those having an OAc group in position 2 of the benzene ring show laser activity different from the others, probably due to the interaction of this group with the π electrons of anthracene. In order to explain these differences, X-ray structural studies of some

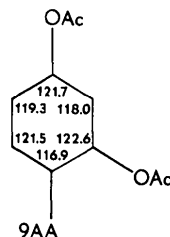
* IUPAC name: 4-(10-acetoxy-9-anthryl)-1,3-phenylene diacetate.

9-acetoxy-10-arylanthracenes were undertaken. Another reason was to determine the influence of substituents, mainly OAc groups, on the aromatic systems. For the title compound (ADA hereafter) the analysis proposed by Domenicano, Mazzeo & Vaciago (1976) and Domenicano & Murray-Rust (1979, hereafter DMR) can be applied.



Experimental. ADA prepared by Hubacher's (1958) method; light-brown parallelepipedal crystals grown from ethanol by slow evaporation; crystal system and approximate cell dimensions determined from Weissenberg and oscillation photographs; density measured by flotation in aqueous KI solution; crystal specimen $0.2 \times 0.3 \times 0.5$ mm; Syntex $P2_1$ four-circle diffractometer, graphite-monochromated $\text{Cu K}\alpha$ radiation, θ - 2θ scan mode, background and intensity of reflections calculated by peak-profile analysis (Lehmann & Larsen, 1974; Jaskólski, 1979); accurate cell parameters refined from setting angles of 15 reflections with $10 < \theta < 26^\circ$; max. $(\sin \theta) / \lambda = 0.5460 \text{ \AA}^{-1}$, $-11 \leq h \leq 11$, $-15 \leq k \leq 15$, $0 \leq l \leq 9$, 2834 unique reflections measured, 2467 considered observed [$I > 1.96 \sigma(I)$], L_p correction but no absorption correction. Structure solved by direct methods with *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), renormalization of E 's using smaller temperature factor $B = 3.0$ instead of $B = 3.6 \text{ \AA}^2$ calculated by program was necessary, best set of phases allowed location of all non-H atoms; choice of space group $P\bar{1}$ justified by calculated E statistics and successful refinement. Structure refined by full-matrix least squares with *SHELX76* (Sheldrick, 1976), function minimized $\sum w(|F_o| - |F_c|)^2$, $w^{-1} = \sigma^2(F_o)$, $\sigma(F_o)$ based on counting statistics; 13 H atoms located from difference synthesis, positions of remaining H atoms calculated, CH_3 groups treated initially as rigid groups with tetrahedral geometry and constrained C-H distances of 1.08 \AA , finally all constraints removed; anisotropic thermal parameters for non-H atoms and isotropic for H atoms; two very strong reflections appeared to suffer from extinction and were omitted; final $R = 0.042$, $wR = 0.052$ and $S = 5.66$; max. and mean Δ/σ in last cycle 0.39 and 0.02 ; max. and min. height in final $\Delta\rho$ map $+0.18$ and -0.21 e \AA^{-3} ; atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. The positional parameters and U_{eq} for non-H atoms are given in Table 1.* A stereoview of the molecule with atom labelling is shown in Fig. 1. Bond lengths and angles are listed in Table 2. In order to explain the values of the internal angles of the benzene ring, DMR analysis was used. In accordance with the suggestion of Domenicano & Murray-Rust (1979) this method can be applied not only to mono- and *para*-disubstituted but also to polysubstituted benzene derivatives. ADA was therefore treated as a tri-substituted benzene derivative in which each substituent, two OAc groups and one 9-acetoxy-10-anthryl (9AA hereafter), induces distortions $\Delta\alpha$, $\Delta\beta$, $\Delta\gamma$ and $\Delta\delta$ from 120° of all internal angles of the ring (idealized $mm2$ symmetry for the substituent effects is used).



With the assumption that substituent effects are independent and can be superimposed, the following angular parameters were calculated (using additionally the DMR experimental equation $\Delta\beta = -0.54\Delta\alpha - 0.30$):

Substituent	$\Delta\alpha$	$\Delta\beta$	$\Delta\gamma$	$\Delta\delta$
OAc	1.4	-1.1	0.3	0.2
9AA	-2.2	0.9	0.2	0.0

With Taft's inductive parameter, σ_p , for the OAc group taken as 0.42 (Brownlee, Hutchinson, Katritzky, Tidwell & Topsom, 1968), the OAc group can be included in the DMR correlation diagram of $\Delta\alpha$ vs σ_p . Fig. 2 shows that the OAc group, having a weak σ -electron-withdrawing character, fits well this correlation line. The angular parameters for OAc and 9AA must, however, be confirmed by data from other compounds. For this purpose the structural analysis of *p*-diacetoxybenzene has been carried out (Roszak, Borowiak & Gawron, 1984) giving for the OAc group $\Delta\alpha + \Delta\delta = 1.7$ and $\Delta\beta + \Delta\gamma = -0.85$, which are in a good agreement with the results of this work. Additionally, similar analysis for some other *para*-disubstituted benzene derivatives (with OAc as one of the substituents) and 9-acetoxy-10-arylanthracenes will be carried out.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond lengths and angles involving H atoms, least-squares-planes' data and a *PLUTO* (Motherwell & Clegg, 1978) drawing of the molecular packing have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42580 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The internal angles at the C atom to which the OAc group is bonded are: 121.0 (3)° for acetylsalicylic acid (Wheatley, 1964), 122.9 (3)° for 2-acetoxy-3-methylbenzoic acid (Chiari, Fronczek, Davis & Gandour, 1981) and 122.2 (1)° for 2-acetoxy-6-methylbenzoic acid (Fronczek, Merrill & Gandour, 1982) and they also correspond to the σ -electron-withdrawing character of this substituent. However, DMR analysis for the first two of these, where the COOH group is almost coplanar with the benzene ring, assuming known

Table 1. Fractional coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^4$) for non-H atoms with e.s.d.'s in parentheses

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
C(1)	7650 (3)	3044 (2)	5540 (3)	351 (7)
C(2)	7313 (3)	2838 (2)	3702 (3)	358 (7)
C(3)	6019 (3)	3072 (2)	2287 (4)	435 (8)
C(4)	5689 (3)	2856 (2)	515 (3)	521 (9)
C(5)	6624 (3)	2386 (2)	-0 (4)	539 (10)
C(6)	7868 (3)	2153 (2)	1285 (4)	473 (9)
C(7)	8263 (3)	2367 (2)	3177 (3)	385 (7)
C(8)	9516 (3)	2138 (2)	4546 (3)	403 (8)
C(9)	9941 (3)	2386 (2)	6403 (3)	402 (8)
C(10)	11287 (3)	2187 (2)	7819 (4)	510 (9)
C(11)	11662 (3)	2443 (2)	9609 (4)	596 (10)
C(12)	10722 (3)	2891 (2)	10133 (4)	578 (10)
C(13)	9419 (3)	3078 (2)	8826 (4)	465 (9)
C(14)	8981 (3)	2850 (2)	6920 (3)	376 (7)
C(15)	6549 (3)	3415 (2)	5998 (3)	359 (7)
C(16)	5118 (3)	2810 (2)	5195 (3)	362 (7)
C(17)	4095 (3)	3108 (2)	5651 (4)	426 (8)
C(18)	4513 (3)	4048 (2)	6932 (3)	416 (8)
C(19)	5883 (3)	4684 (2)	7713 (4)	438 (9)
C(20)	6896 (3)	4365 (2)	7248 (4)	439 (8)
C(21)	9976 (4)	645 (2)	3252 (5)	641 (12)
C(22)	11116 (6)	212 (4)	2937 (10)	942 (20)
C(23)	5311 (3)	1143 (2)	4163 (4)	494 (9)
C(24)	4914 (7)	348 (3)	2408 (7)	782 (16)
C(25)	2187 (3)	4479 (2)	6316 (4)	460 (9)
C(26)	1401 (4)	4854 (3)	7277 (5)	529 (11)
O(1)	10451 (2)	1656 (1)	4055 (3)	494 (6)
O(2)	8803 (3)	202 (2)	2874 (4)	1036 (11)
O(3)	4614 (2)	1901 (1)	3742 (2)	436 (5)
O(4)	6129 (2)	1161 (1)	5744 (3)	622 (7)
O(5)	3579 (2)	4358 (1)	7375 (2)	510 (6)
O(6)	1747 (2)	4314 (2)	4715 (3)	737 (9)

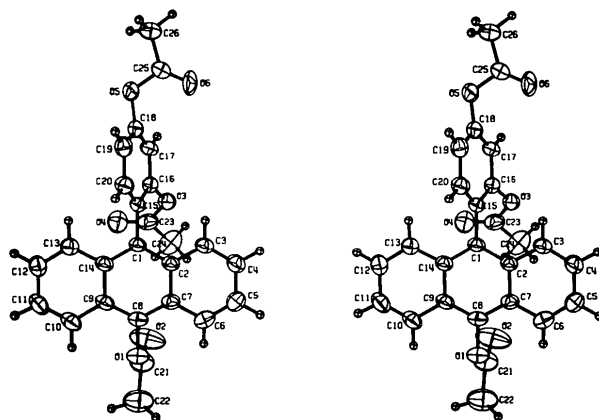


Fig. 1. Stereoview of the ADA molecule with 50% probability thermal ellipsoids (ORTEPII; Johnson, 1976).

substituent effects of Me and COOH (Domenicano & Murray-Rust, 1979), is not unequivocal. In the third structure, the COOH group is twisted by 44.7° from the plane of the benzene ring and, in this case, the

Table 2. Bond lengths (\AA) and angles ($^\circ$) with e.s.d.'s in parentheses

C(1)—C(2)	1.403 (3)	C(15)—C(16)	1.395 (3)
C(1)—C(14)	1.409 (3)	C(16)—C(17)	1.381 (3)
C(2)—C(3)	1.430 (3)	C(16)—O(3)	1.399 (3)
C(2)—C(7)	1.434 (3)	C(17)—C(18)	1.380 (3)
C(3)—C(4)	1.349 (3)	C(18)—C(19)	1.369 (4)
C(4)—C(5)	1.415 (4)	C(18)—O(5)	1.403 (3)
C(5)—C(6)	1.352 (4)	C(19)—C(20)	1.384 (3)
C(6)—C(7)	1.428 (3)	C(15)—C(20)	1.392 (3)
C(7)—C(8)	1.386 (3)	C(21)—O(1)	1.365 (3)
C(8)—C(9)	1.385 (3)	C(21)—O(2)	1.186 (3)
C(8)—O(1)	1.411 (3)	C(21)—C(22)	1.496 (5)
C(9)—C(10)	1.434 (3)	C(23)—O(3)	1.371 (3)
C(10)—C(11)	1.345 (4)	C(23)—O(4)	1.192 (3)
C(11)—C(12)	1.411 (4)	C(23)—C(24)	1.488 (4)
C(12)—C(13)	1.362 (4)	C(25)—O(5)	1.365 (3)
C(13)—C(14)	1.424 (3)	C(25)—O(6)	1.180 (3)
C(9)—C(14)	1.435 (3)	C(25)—C(26)	1.489 (4)
C(1)—C(15)	1.492 (3)		
C(2)—C(1)—C(14)	120.2 (2)	C(1)—C(15)—C(16)	120.8 (2)
C(2)—C(1)—C(15)	119.7 (2)	C(1)—C(15)—C(20)	122.3 (2)
C(14)—C(1)—C(15)	120.0 (2)	C(20)—C(15)—C(16)	116.9 (2)
C(1)—C(2)—C(3)	122.0 (2)	C(15)—C(16)—C(17)	122.6 (2)
C(1)—C(2)—C(7)	120.4 (2)	C(15)—C(16)—O(3)	119.9 (2)
C(7)—C(2)—C(3)	117.6 (2)	C(17)—C(16)—O(3)	117.2 (2)
C(2)—C(3)—C(4)	121.6 (2)	C(16)—C(17)—C(18)	118.0 (2)
C(3)—C(4)—C(5)	120.6 (3)	C(17)—C(18)—C(19)	121.7 (2)
C(4)—C(5)—C(6)	120.3 (3)	C(17)—C(18)—O(5)	120.4 (2)
C(5)—C(6)—C(7)	121.2 (3)	C(19)—C(18)—O(5)	117.7 (2)
C(6)—C(7)—C(8)	123.6 (2)	C(18)—C(19)—C(20)	119.3 (2)
C(2)—C(7)—C(6)	118.7 (2)	C(19)—C(20)—C(15)	121.5 (3)
C(2)—C(7)—C(8)	117.7 (2)	C(8)—O(1)—C(21)	115.6 (2)
C(7)—C(8)—C(9)	123.6 (2)	O(1)—C(21)—C(22)	110.8 (3)
C(7)—C(8)—O(1)	118.8 (2)	O(1)—C(21)—O(2)	122.4 (3)
C(9)—C(8)—O(1)	117.5 (2)	O(2)—C(21)—C(22)	126.8 (3)
C(8)—C(9)—C(10)	122.6 (2)	C(16)—O(3)—C(23)	118.7 (2)
C(8)—C(9)—C(14)	118.4 (2)	O(3)—C(23)—C(24)	110.6 (3)
C(10)—C(9)—C(14)	119.0 (2)	O(3)—C(23)—O(4)	122.5 (2)
C(9)—C(10)—C(11)	120.8 (3)	O(4)—C(23)—C(24)	127.0 (3)
C(10)—C(11)—C(12)	120.9 (3)	C(18)—O(5)—C(25)	118.3 (2)
C(11)—C(12)—C(13)	120.2 (3)	O(5)—C(25)—C(26)	110.4 (2)
C(12)—C(13)—C(14)	121.6 (3)	O(5)—C(25)—O(6)	121.9 (2)
C(13)—C(14)—C(1)	123.0 (2)	O(6)—C(25)—C(26)	127.7 (3)
C(13)—C(14)—C(9)	117.5 (2)		
C(9)—C(14)—C(1)	119.5 (2)		

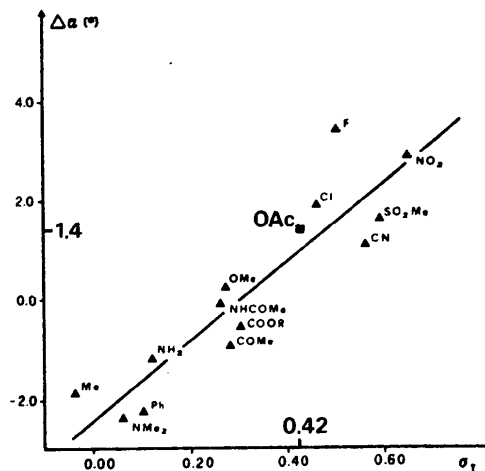
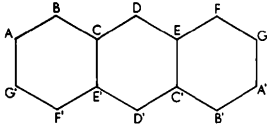


Fig. 2. Domenicano & Murray-Rust (1979) plot of the substituent parameter $\Delta\sigma$ vs σ_I with our data for the OAc group included.

Table 3. A comparison of bond lengths (Å) and angles (°) for the anthracene moiety of ADA with those given by Harlow *et al.* (1975) for 'average unsubstituted' and 'average 9,10-*sp*²-disubstituted' anthracene



	Average unsubstituted anthracene*	Average 9,10- <i>sp</i> ² -disubstituted anthracene*	This work†
AB	1.367	1.354	1.352 (4)
BC	1.433	1.432	1.429 (2)
CD	1.399	1.402	1.406 (3)
C'D'			1.386 (1)
AG'	1.419	1.413	1.413 (2)
CE'	1.433	1.438	1.435 (1)
G'AB	120.8	120.4	120.5 (2)
ABC	120.3	121.4	121.3 (2)
BCE'	118.9	118.0	117.6 (1)
B'C'E			118.9 (2)
BCD	121.7	122.2	122.8 (3)
E'CD	119.5	119.8	120.0 (5)
EC'D'			118.1 (4)
CDE	121.0	120.5	120.2 (2)
C'D'E'			123.6 (2)

* *mmm* symmetry assumed.

† *mmm* symmetry assumed except for those values for which differences are significant according to the Cruickshank & Robertson (1953) test, then *mm2* symmetry assumed.

analysis of angular deviations from 120° gives satisfactory results if the angular substituent parameters are those of Norrestam & Schepper (1981) for non-coplanar COOH. A probable reason for this could be the influence of even small steric hindrance of the COOH group, coplanar with the benzene ring, on the deviations from additivity of substituent effects, when *ortho* substituents are present in the molecule. This is in agreement with the more general interpretation given by Colapietro, Domenicano & Marciante (1982) of deviations from additivity in *ortho*-disubstituted benzene derivatives. The other structure with an OAc group, 1-acetoxy-4-nitrobenzene (*p*-nitrophenyl acetate) (Guttormson & Robertson, 1972), could not be analysed in this way, as the accuracy of this determination is too low for such calculations.

A comparison of bond lengths and angles for the anthracene moiety found here with those of 'average unsubstituted' and '9,10-*sp*²-disubstituted' anthracene (Harlow, Loghry, Williams & Simonsen, 1975) is shown in Table 3. The influence of the OAc group bonded to atom D' on the geometry of anthracene is clear: the C'D'E' angle is increased by 2.6 (2)°, the angles EC'D' are decreased by 1.4 (4)° and the bonds C'D' are contracted by 0.013 (1) Å. The directions of these changes are in agreement with DMR-analysis results for OAc and with the general theory of such substitution effects (Domenicano, Vaciago & Coulson,

1975). The effect of the 2,4-diacetoxyphenyl substituent is not so large; changes for CDE, E'CD, CD and BCE' are: -0.8 (2), +0.5 (5)°, +0.007 (3) Å and -1.3 (1)°, *i.e.* they have opposite signs, which agrees with the σ -electron-releasing character of the phenyl group (Domenicano & Murray-Rust, 1979). Similar directions of changes are observed for 'average 9,10-*sp*²-disubstituted' anthracene (Harlow *et al.*, 1975). Additionally, for both 9,10-disubstituted anthracenes (see Table 3), the AB bonds are significantly contracted [by 0.015 (4) Å for ADA], having almost pure double-bond character, which is the most probable reason for the significant increase of angles ABC and BCD [by 1.0 (2) and 1.1 (3)°, respectively, for ADA].

The anthracene ring system is not strictly planar, as in the case of other noncentrosymmetric anthracene derivatives, *e.g.* 9-methylanthracene (Cox & Sim, 1979) and its derivatives (Glusker & Zacharias, 1972; Stallings, Monti & Glusker, 1982) and 9-[(2-chloroethyl)thio]methylanthracene (Lewis, Carrell, Glusker & Sparks, 1976), but is slightly bent along the C(1)-C(8) line as shown in Fig. 3(a). The r.m.s. deviation from the least-squares plane through the 14 atoms of the anthracene is 0.035 Å. The outer rings are almost planar and form a dihedral angle of 2.9° (*e.s.d.*'s for dihedral angles are 0.3°).

The bonds C(1)-C(15) and C(8)-O(1), connecting the anthracene ring system with its 9- and 10-substituents, deviate from the least-squares plane of the anthracene moiety in a direction opposite to that of the direction of bending [Fig. 3(a)], which minimizes the steric interactions. Fig. 3(b) shows the values of these angle deviations. The displacements from the anthracene plane are 0.217 (2) Å for C(15) and 0.089 (2) Å for O(1).

The least-squares plane of the benzene ring forms a dihedral angle of 68.2° with the anthracene least-squares plane.

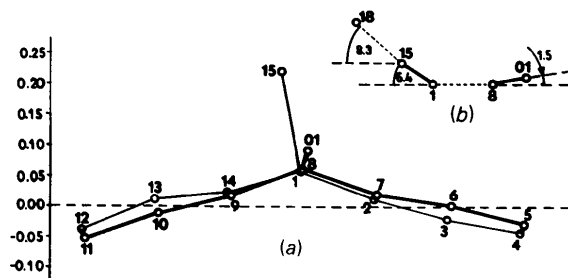


Fig. 3. (a) Distortions (Å) in the anthracene ring system in relation to its least-squares plane. The vertical scale is enlarged tenfold. (b) Angle deviations (°) of bonds C(1)-C(15) and C(8)-O(1) and the axis of the phenyl ring from the least-squares plane of anthracene. The angles are enlarged fivefold in the figure. *E.s.d.*'s are 0.1-0.2°.

All the acetoxy groups are planar and their geometry is similar to that reported earlier (*e.g.* Foces-Foces, Cano & García-Blanco, 1980). The OAc groups bonded to the benzene ring at C(16) and C(18) form dihedral angles of 75.6 and 63.8°, respectively, with the least-squares plane of the ring. The displacements of O(3) and O(5) from the benzene-ring plane are 0.175 (2) and 0.142 (2) Å, respectively. The OAc group bonded to anthracene forms a dihedral angle of 81.8° with the anthracene least-squares plane.

The geometry of the anthracene ring system is, in general, very similar to that of 9-acetoxy-10-(2-acetoxy-5-bromophenyl)anthracene (Roszak & Skrzat, 1985).

There are no unusually short contacts in this structure.

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References

- BROWNLEE, R. T. C., HUTCHINSON, R. E. J., KATRITZKY, A. R., TIDWELL, T. T. & TOPSOM, R. D. (1968). *J. Am. Chem. Soc.* **90**, 1757–1767.
- CHIARI, G., FRONCZEK, F. R., DAVIS, S. T. & GANDOUR, R. D. (1981). *Acta Cryst.* **B37**, 1623–1625.
- COLAPIETRO, M., DOMENICANO, A. & MARCIANTE, C. (1982). *Annals of the Yugoslav Centre of Crystallography*, Supplement to Vol. 17, pp. S140–S142. Zagreb: Yugoslav Academy of Sciences and Arts.
- COX, P. J. & SIM, G. A. (1979). *Acta Cryst.* **B35**, 404–410.
- CRUICKSHANK, D. W. J. & ROBERTSON, A. P. (1953). *Acta Cryst.* **6**, 698–705.
- DOMENICANO, A., MAZZEO, P. & VACIAGO, A. (1976). *Tetrahedron Lett.* pp. 1029–1032.
- DOMENICANO, A. & MURRAY-RUST, P. (1979). *Tetrahedron Lett.* pp. 2283–2286.
- DOMENICANO, A., VACIAGO, A. & COULSON, C. A. (1975). *Acta Cryst.* **B31**, 221–234.
- FOCES-FOCES, C., CANO, F. H. & GARCÍA-BLANCO, S. (1980). *Acta Cryst.* **B36**, 377–384.
- FRONCZEK, F. R., MERRILL, M. L. & GANDOUR, R. D. (1982). *Acta Cryst.* **B38**, 1337–1339.
- GLUSKER, J. P. & ZACHARIAS, D. E. (1972). *Acta Cryst.* **B28**, 2702–2708.
- GRONOWSKA, J., ALEKSANDRZAK, K. & HELDT, J. (1981). *Pol. J. Chem.* **55**, 57–65.
- GUTTORMSON, R. & ROBERTSON, B. E. (1972). *Acta Cryst.* **B28**, 2702–2708.
- HARLOW, R. L., LOGHRY, R. A., WILLIAMS, H. J. & SIMONSEN, S. H. (1975). *Acta Cryst.* **B31**, 1344–1350.
- HELDT, J. R. (1983). *Z. Naturforsch. Teil A*, **38**, 1197–1205.
- HUBACHER, M. H. (1958). *J. Org. Chem.* **23**, 1400–1402.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- JASKÓLSKI, M. (1979). Thesis, A. Mickiewicz Univ., Poznań, Poland.
- JOHNSON, C. K. (1976). *ORTEP*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.
- LEHMANN, M. S. & LARSEN, F. K. (1974). *Acta Cryst.* **A30**, 580–584.
- LEWIS, M., CARRELL, H. L., GLUSKER, J. P. & SPARKS, R. A. (1976). *Acta Cryst.* **B32**, 2040–2044.
- MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MOTHERWELL, W. D. S. & CLEGG, P. (1978). *PLUTO*. A program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- NORRESTAM, R. & SCHEPPER, L. (1981). *Acta Chem. Scand. Ser. A*, **35**, 91–103.
- ROZAK, A., BOROWIAK, T. & GAWRON, M. (1984). *Acta Cryst.* **A40**, C100.
- ROZAK, A. & SKRZAT, Z. (1985). *Acta Cryst.* **C41**, 1483–1486.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- STALLINGS, W. C., MONTI, C. T. & GLUSKER, J. P. (1982). *Acta Cryst.* **B38**, 177–184.
- WHEATLEY, P. J. (1964). *J. Chem. Soc.* pp. 6036–6048.

Acta Cryst. (1986). **C42**, 347–349

Structure of 2-(4-Chlorophenylhydrazono)-N-methylacetoacetamide

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Abstract. $C_{11}H_{12}ClN_3O_2$, $M_r = 253.7$, $P2_1/a$, $a = 11.476$ (1), $b = 13.918$ (3), $c = 7.565$ (5) Å, $\beta = 97.9$ (1)°, $V = 1196.8$ Å³, $Z = 4$, $D_x = 1.41$, $D_m = 1.39$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 2.65$ cm⁻¹, $F(000) = 528$, room temperature, $R = 0.0558$ with

1002 independent non-zero reflections. Intermolecular bonding is entirely by van der Waals interactions. However, the molecule is stabilized by intramolecular hydrogen bonds. Except for one, all the bond lengths agree with those reported for azo pigments. The