C2	0.6197 (9)	0.3194 (2)	0.3173 (8)	0.047 (3)
C3	0.6646 (9)	0.3728 (3)	0.3125 (8)	0.048 (3)
C4	0.495 (1)	0.4047 (2)	0.2295 (8)	0.049 (3)
C5	0.2889 (10)	0.3863 (2)	0.1550 (8)	0.050 (3)
C6	0.2447 (9)	0.3327 (2)	0.1631 (8)	0.046 (3)
C7	0.5193 (9)	0.2086 (2)	0.2211 (8)	0.043 (3)
C8	0.4902 (9)	0.1517 (2)	0.2271 (7)	0.037 (2)
C9	0.6765 (9)	0.1222 (2)	0.1550 (8)	0.048 (3)
C10	0.668 (1)	0.0679 (2)	0.1543 (9)	0.055 (3)
C11	0.475 (1)	0.0417 (3)	0.2283 (9)	0.057 (3)
C12	0.2919 (10)	0.0708 (3)	0.3028 (9)	0.055 (3)
C13	0.2973 (9)	0.1250 (2)	0.3022 (8)	0.045 (3)
F4	0.5392 (6)	0.4577 (1)	0.2259 (5)	0.074 (2)

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

O1—N1	1.300 (5)	C5—C6	1.381 (9)
N1—C1	1.463 (6)	C7—C8	1.450 (8)
N1—C7	1.298 (6)	C8—C9	1.392 (7)
C1—C2	1.373 (7)	C8—C13	1.393 (7)
C1—C6	1.369 (7)	C9—C10	1.373 (8)
C2—C3	1.373 (8)	C10—C11	1.383 (8)
C3—C4	1.378 (8)	C11—C12	1.382 (8)
C4—C5	1.351 (8)	C12—C13	1.371 (8)
C4—F4	1.363 (7)		
O1—N1—C1	115.1 (4)	N1—C1—C6	118.0 (5)
O1—N1—C7	124.7 (4)	N1—C7—C8	126.1 (5)
C1—N1—C7	120.2 (4)	C7—C8—C9	115.9 (5)
N1—C1—C2	120.9 (5)	C7—C8—C13	125.6 (5)

Data collection: *CAD*-4 *Operations Manual* (Enraf–Nonius, 1977). Cell refinement: *CAD*-4 *Operations Manual* (Enraf–Nonius, 1977). Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN FINISH*. Literature Survey: *CSSR* (1984).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including H-atom geometry, have been deposited with the IUCr (Reference: HU1138). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5,5′-Di(anthracenecarboxylic) Anhydride

Alexander J. Blake, Rachel S. Grimditch and Martin Schröder

Department of Chemistry, The University of Edinburgh, West Mains Road, Edinburgh EH9 3JJ, Scotland

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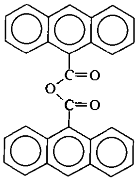
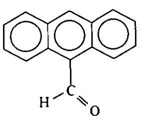
Abstract

The title compound, $C_{30}H_{18}O_3$, has a dihedral angle of 76.24 (5)° between the two anthracene units and a mean dihedral angle of 64.0 (2)° between an anthracene unit and the plane defined by C5 and the C and O atoms of the carbonyl group.

Comment

It has been shown that in 5-substituted anthracenes the torsion angle between the aromatic π system and the substituent is governed by steric requirements (Trotter, 1959). The least bulky anthracene carbonyl derivative, 5-anthraldehyde, (2), exhibits a torsion of 27° (Trotter, 1959), while the maximum value of 88° is found for 5-acetylanthracene, (3) (Andersson, Becker, Engelhardt, Hansen & White, 1984), with torsion angles for related molecules falling between these values. The value of 64.0 (2)° for the title compound, (1), compares with those of 50.5° for dianthril ketone, (4) (Becker, Langer, Skelton & White, 1989), 75.0° for 5,5-anthroin acetate, (6) (Becker, Kildea, Patrick, Skelton & White, 1991), and 54.0 and 86.15° for the α (5α) and β (5β) forms, respectively, of 5,5′-anthril (Becker, Skelton & White, 1991).


(1)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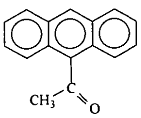
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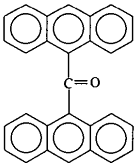
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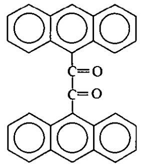
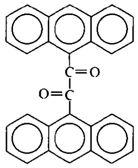
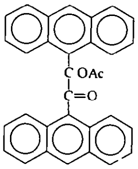

(5α)


(5β)


(6)

The dihedral angle between the two anthracene units is also of interest, the value of 76.24 (5)° in (1) falling between those of 87.3° in (4) and 74.8° in (5α).

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Typical values for this parameter lie in the range 60–80°, the exception being that of the β form of 5,5-anthril (5β) (Becker, Skelton & White, 1991).

Both the C—O [1.384 (2), 1.390 (2) Å] and C═O [1.187 (2), 1.184 (2) Å] distances in (1) lie in the expected range for acid anhydrides (Allen *et al.*, 1987). As has been found previously, the presence of the carbonyl group on C5 leads to marked asymmetry in the central ring of each anthracene moiety: C—C bonds involving C5 are significantly longer than those involving C10.

The molecular packing (Fig. 2) exhibits several interesting features. The molecules are stacked in sheets which lie in the *bc* plane; these sheets repeat along the *a* axis. Within a stack, each anthracene ring lies parallel to one ring of a neighbouring molecule and perpendicular both to that molecule's other ring and to a ring from a neighbouring molecule. Parallel rings are well separated at 4.340 Å.

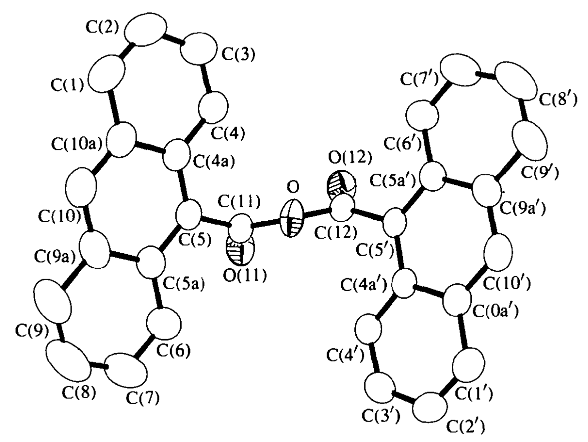

Fig. 1. A view of a molecule of (1) with atom-numbering scheme. Displacement ellipsoids enclose 50% probability surfaces; H atoms are omitted for clarity. Note that the substituted C atom on the central anthracene ring is C5, rather than C9 as was the convention previously.

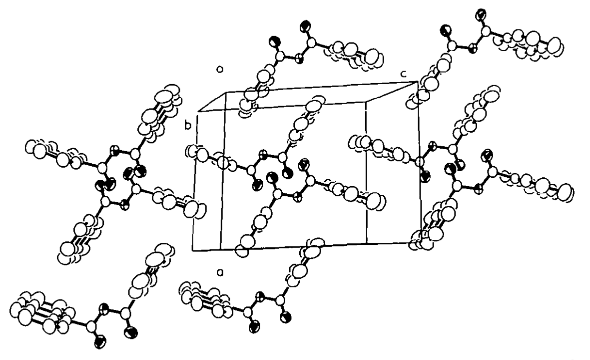

Fig. 2. A packing diagram showing part of a two-dimensional stacked sheet of molecules of (1). H atoms are omitted for clarity.

Experimental

Treatment of 5-anthracenecarboxylic acid with oxalyl chloride yields 5-anthracenecarboxyl chloride, which reacts with residual acid to give 5,5′-di(anthracenecarboxylic) anhydride, (1). Crystals of (1) were grown by the slow concentration of a dichloromethane solution.

Crystal data

$C_{30}H_{18}O_3$
$M_r = 426.44$
Triclinic
$P\bar{1}$
$a = 9.834 (5)$ Å
$b = 10.973 (6)$ Å
$c = 11.510 (6)$ Å
$\alpha = 112.51 (2)°$
$\beta = 89.56 (2)°$
$\gamma = 107.24 (2)°$
$V = 1087.9 (10)$ Å^3
$Z = 2$
$D_x = 1.302$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 12 reflections
$\theta = 15–16°$
$\mu = 0.083$ mm^{-1}
$T = 295 (2)$ K
Lath
0.82 × 0.39 × 0.19 mm
Yellow

Data collection

Stoe Stadi-4 four-circle diffractometer
$\omega–2\theta$ scans using on-line profile-fitting (Clegg, 1981)
Absorption correction: none
6230 measured reflections
6230 independent reflections

4003 observed reflections [$I > 2\sigma(I)$]
$\theta_{\max} = 30.02°$
$h = -13 \rightarrow 13$
$k = -15 \rightarrow 14$
$l = 0 \rightarrow 16$
3 standard reflections
frequency: 60 min
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0555$
$wR(F^2) = 0.1285$
$S = 1.070$
6099 reflections
299 parameters
H atoms included in calculated positions and allowed to ride on the corresponding C atoms with $U_{\rm iso} = 1.2U_{\rm eq}({\rm C})$
$w = 1/[\sigma^2(F_o^2) + (0.0557P)^2 + 0.3047P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.017$
$\Delta\rho_{\max} = 0.222$ e Å^{-3}
$\Delta\rho_{\min} = -0.169$ e Å^{-3}
Extinction correction: *SHELXL93* (Sheldrick, 1993)
Extinction coefficient: 0.0064 (19)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	$U_{\rm eq}$
C11	0.6365 (2)	0.8355 (2)	0.45459 (14)	0.0446 (4)
O11	0.52184 (14)	0.8336 (2)	0.42084 (13)	0.0637 (4)
O	0.66610 (12)	0.82598 (13)	0.56761 (10)	0.0520 (3)
C12	0.5608 (2)	0.7589 (2)	0.62373 (14)	0.0429 (3)
O12	0.45730 (14)	0.66601 (13)	0.56461 (11)	0.0615 (4)
C1	1.0130 (2)	0.6592 (2)	0.2274 (2)	0.0693 (6)
C2	0.9518 (3)	0.5356 (2)	0.2357 (2)	0.0749 (6)
C3	0.8282 (2)	0.5145 (2)	0.2974 (2)	0.0660 (5)
C4	0.7682 (2)	0.6164 (2)	0.3485 (2)	0.0531 (4)

C4*A*	0.8288 (2)	0.7492 (2)	0.34266 (14)	0.0440 (4)
C5	0.7691 (2)	0.8574 (2)	0.39180 (14)	0.0425 (3)
C5*A*	0.8297 (2)	0.9861 (2)	0.38296 (15)	0.0455 (4)
C6	0.7750 (2)	1.1001 (2)	0.4369 (2)	0.0584 (5)
C7	0.8397 (2)	1.2230 (2)	0.4273 (2)	0.0735 (6)
C8	0.9614 (2)	1.2409 (3)	0.3626 (3)	0.0801 (7)
C9	1.0170 (2)	1.1365 (2)	0.3106 (2)	0.0703 (6)
C9*A*	0.9558 (2)	1.0058 (2)	0.3202 (2)	0.0522 (4)
C10	1.0144 (2)	0.8983 (2)	0.2712 (2)	0.0572 (5)
C10*A*	0.9550 (2)	0.7713 (2)	0.2804 (2)	0.0508 (4)
C1′	0.6942 (2)	1.1525 (2)	1.0439 (2)	0.0601 (5)
C2′	0.6544 (2)	1.2326 (2)	0.9959 (2)	0.0642 (5)
C3′	0.5963 (2)	1.1773 (2)	0.8674 (2)	0.0583 (5)
C4′	0.5812 (2)	1.0433 (2)	0.7901 (2)	0.0480 (4)
C4*A*′	0.6228 (2)	0.9548 (2)	0.83671 (14)	0.0410 (3)
C5′	0.6082 (2)	0.8151 (2)	0.76153 (13)	0.0399 (3)
C5*A*′	0.6443 (2)	0.7283 (2)	0.81138 (15)	0.0438 (3)
C6′	0.6354 (2)	0.5876 (2)	0.7366 (2)	0.0554 (4)
C7′	0.6747 (3)	0.5099 (2)	0.7889 (2)	0.0722 (6)
C8′	0.7240 (3)	0.5658 (2)	0.9195 (2)	0.0850 (7)
C9′	0.7353 (3)	0.6984 (2)	0.9934 (2)	0.0725 (6)
C9*A*′	0.6985 (2)	0.7862 (2)	0.9428 (2)	0.0516 (4)
C10′	0.7155 (2)	0.9249 (2)	1.0165 (2)	0.0554 (4)
C0*A*′	0.6794 (2)	1.0108 (2)	0.96776 (15)	0.0477 (4)

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

C11—O11	1.187 (2)	C5*A*—C9*A*	1.433 (2)
C11—O	1.384 (2)	C9*A*—C10	1.390 (3)
C11—C5	1.488 (2)	C10—C10*A*	1.387 (3)
O—C12	1.390 (2)	C4*A*′—C5′	1.406 (2)
C12—O12	1.184 (2)	C4*A*′—C0*A*′	1.436 (2)
C12—C5′	1.485 (2)	C5′—C5*A*′	1.408 (2)
C4*A*—C5	1.403 (2)	C5*A*′—C9*A*′	1.433 (2)
C4*A*—C10*A*	1.432 (2)	C9*A*′—C10′	1.387 (3)
C5—C5*A*	1.403 (2)	C10′—C0*A*′	1.392 (3)
O11—C11—O	122.47 (15)	O—C12—C5′	108.72 (13)
O11—C11—C5	127.5 (2)	C5*A*—C5—C11	118.50 (15)
O—C11—C5	109.94 (13)	C4*A*—C5—C11	119.31 (15)
C11—O—C12	122.36 (13)	C4*A*′—C5′—C12	119.09 (14)
O12—C12—O	122.74 (15)	C5*A*′—C5′—C12	118.74 (14)
O12—C12—C5′	128.3 (2)		
O11—C11—O—C12	27.0 (3)	O11—C11—C5—C4*A*	−115.3 (2)
C5—C11—O—C12	−156.75 (14)	O—C11—C5—C4*A*	68.7 (2)
C11—O—C12—O12	28.5 (2)	O12—C12—C5′—C4*A*′	−119.4 (2)
C11—O—C12—C5′	−156.46 (14)	O—C12—C5′—C4*A*′	65.9 (2)
O11—C11—C5—C5*A*	64.2 (2)	O12—C12—C5′—C5*A*′	63.8 (2)
O—C11—C5—C5*A*	−111.8 (2)	O—C12—C5′—C5*A*′	−110.9 (2)

Data collection: *DIF*4 (Stoe & Cie, 1990*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1990*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1992).

We thank EPSRC for support.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: AB1274). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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