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2-(*N*,*N*-Diacetamido)-3a,4,9,9a-tetrahydro-4,9-[1′,2′]benzeno-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.138
Data-to-parameter ratio = 12.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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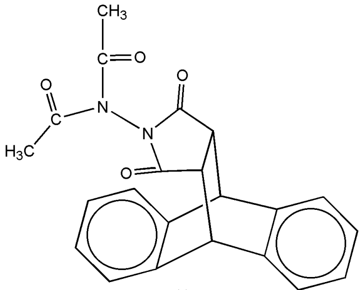
The title compound, $C_{22}H_{18}N_2O_4$, has a molecular structure with a rigid polycyclic nucleus, in which a dicarboximide ring bridges the two CH positions of the central ring of anthracene. The *N*-(*N*′-diacetyl)succinimide system is oriented to one side of the molecule. The *N*′-diacetyl fragment and the dicarboximide ring planes are almost perpendicular. Short intermolecular C—H···O contacts are observed.

Comment

The Diels–Alder reaction of anthracene and maleimide derivatives is recently of growing interest. A series of the products were efficiently produced using artificial ribozymes as the catalysts (Stuhlmann & Jaschke, 2002, and references therein). Anthracene-tagged substrates are employed as protecting groups in conjunction with a polymer-bound maleimide dienophile (Wang *et al.*, 2000; Lan *et al.*, 2003). The title compound, (I), was obtained unexpectedly as a product of acetylation of the hydrazide synthesized from the Diels–Alder adduct of anthracene and maleimide (Srivastava *et al.*, 1994; Korsch & Riggs, 1966).



(I)

The molecular structure of this compound, showing the atom-labelling scheme, is presented in Fig. 1. A search of the Cambridge Structural Database (CSD, Version 5.24 of November 2002 and three updates; Allen, 2002) reveals 255 structures in which a bridge of two C atoms is added across the opposite CH groups of the central ring of anthracene. In 25 structures, the bridge is in a dicarboximide ring, as in (I), although many of these include different solvates or cocrystals of the same compound (Weber *et al.*, 1991, 1994, 1995; Kishikawa *et al.*, 1997; Kim *et al.*, 2001; Clegg & Harrington, 2002; Csoregh *et al.*, 2002). For these molecules, the rigidity of the polycyclic core is demonstrated by the small range of dihedral angles found between pairs of mean planes defined as follows: plane 1 is the dicarboximide ring; plane 2 is the C7/C10/C15/C16 plane (using the atom numbering for the title compound; see Fig. 1); plane 3 is C7/C10/C5/C6; and plane 4 is C7/C10/C8/C9. The corresponding values for (I) are 121.76 (8) (1 and 2),

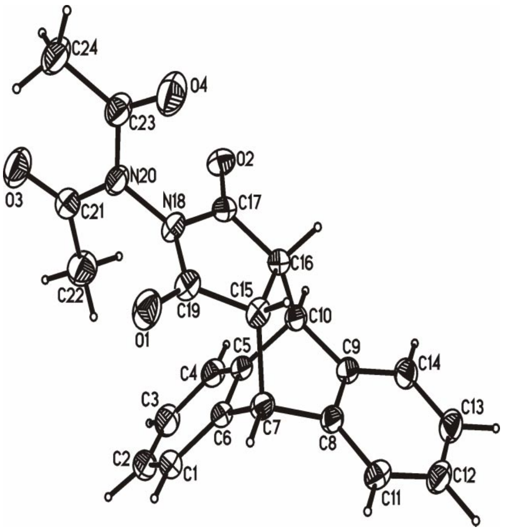


Figure 1
A view of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

119.65 (7) (2 and 3), 117.47 (10) (2 and 4), 122.88 (11)° (3 and 4), each of them within the range for the other carboxamide structures (Weber *et al.*, 1991, 1994, 1995; Kishikawa *et al.*, 1997; Kim *et al.*, 2001; Clegg & Harrington, 2002; Csoregh *et al.*, 2002). The difference between the dihedral angles between planes 2 and 3, and planes 2 and 4, is insignificant and indicates that the C7/C10/C15/C16 bridge is essentially symmetric above the anthracene nucleus, despite the orientation of the *N*-(*N*′-diacetyl)succinimide system to one side of the molecule. The benzene rings are essentially coplanar with planes 3 and 4 – the respective dihedral angles are 178.10 (17) and 178.56 (14)° – so the rigid polycyclic core of this molecule has a shape that agrees with earlier observations of related molecules. The *N*′-diacetyl fragment is approximately planar, with no atomic deviation greater than 0.070 (2) Å (for atom N20) from the least-squares plane. The dicarboximide ring is also planar, with deviations from the mean plane ranging from −0.050 (1) Å for atom N18 to 0.035 (1) Å for atom O2. The *N*′-diacetyl fragment and the dicarboximide ring planes are almost perpendicular, the dihedral angle being 84.16 (4)°. In the crystal structure, there is a network of molecules connected by C—H···O hydrogen bonds (Desiraju, 1996). The geometric parameters of all hydrogen bonds are listed in Table 1.

Experimental

The synthesis of the title compound has been described by Srivastava *et al.* (1994). Crystals were grown from xylene solution by slow evaporation.

Crystal data

$C_{22}H_{18}N_2O_4$
$M_r = 374.38$
Monoclinic, $P2_1/n$
$a = 12.5950\ (10)$ Å
$b = 11.5500\ (10)$ Å
$c = 13.938\ (2)$ Å
$\beta = 116.460\ (10)°$
$V = 1815.2\ (4)$ Å^3
$Z = 4$
$D_x = 1.370$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 37 reflections
$\theta = 7.1–25.3°$
$\mu = 0.78$ mm^{-1}
$T = 293\ (2)$ K
Plate, colourless
0.60 × 0.15 × 0.06 mm

Data collection

Kuma KM-4 four-circle diffractometer
$\omega/2\theta$ scans
9752 measured reflections
3689 independent reflections
2369 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.043$
$\theta_{\rm max} = 75.2°$
$h = -14 \rightarrow 14$
$k = -14 \rightarrow 14$
$l = -16 \rightarrow 16$
2 standard reflections every 100 reflections intensity decay: 0.9%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.138$
$S = 1.04$
3689 reflections
308 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0887P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.27$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.28$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0080 (8)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O1$^{\rm i}$	0.99 (3)	2.55 (3)	3.539 (3)	173 (2)
C11—H11···O4$^{\rm ii}$	0.94 (2)	2.67 (2)	3.488 (3)	146 (2)
C7—H7···O4$^{\rm ii}$	0.97 (2)	2.57 (2)	3.468 (3)	154 (2)
C16—H16···O2$^{\rm iii}$	1.01 (2)	2.38 (2)	3.253 (2)	144 (2)
C24—H24A···O1$^{\rm iv}$	0.95 (3)	2.71 (4)	3.600 (3)	157 (3)

Symmetry codes: (i) $2 - x, 1 - y, 1 - z$; (ii) $\frac{1}{2} + x, \frac{3}{2} - y, \frac{1}{2} + z$; (iii) $2 - x, 1 - y, -z$; (iv) $x - \frac{1}{2}, \frac{3}{2} - y, z - \frac{1}{2}$.

Only positional parameters for H atoms were refined [C—H = 0.94 (2)–1.06 (2) Å], with $U_{\rm iso}$(H) values constrained to be 1.2 (1.5 for methyl groups) times $U_{\rm eq}$ of the carrier atom.

Data collection: *KM*-4 *Software*, Version 8.01 (Kuma, 1996); cell refinement: *KM*-4 *Software*, Version 8.01; data reduction: *KM*-4 *Software*, Version 10.04 (Kuma, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *Stereochemical Workstation* (Siemens, 1989); software used to prepare material for publication: *SHELXL*97.

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References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Clegg, W. & Harrington, R. W. (2002). *Acta Cryst.* E**58**, o1413–o1414.
Csoregh, I., Weber, E. & Finge, S. (2002). *Cryst. Eng.* **5**, 59–70.
Desiraju, G. R. (1996). *Acc. Chem. Res.* **29**, 441–449.
Kim, J. H., Hubig, S. M., Lindeman, S. V. & Kochi, J. K. (2001). *J. Am. Chem. Soc.* **123**, 87–95.
Kishikawa, K., Yoshizaki, K., Kohmoto, S., Yamamoto, M., Yamaguchi, K. & Yamada, K. (1997). *J. Chem. Soc. Perkin Trans.* 1, pp. 1233–1239.
Korsch, B. H. & Riggs, N. V. (1966). *Tetrahedron Lett.* pp. 5897.
Kuma (1996). *KM*-4 *Software.* Version 8.01. Kuma Diffraction, Wrocław, Poland.
Kuma (1998). *KM*-4 *Software.* Version 10.04. Kuma Diffraction, Wrocław, Poland.
Lan, P., Porco, J. A. Jr., South, M. S. & Parlow, J. J. (2003). *J. Comb. Chem.* **5**, 660–669.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Siemens (1989). *Stereochemical Workstation Operation Manual.* Release 3.4. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Srivastava, A., Srivastava, V. & Verma, S. M. (1994). *Pol. J. Chem.* **68**, 29–35.
Stuhlmann, F. & Jaschke, A. J. (2002). *J. Am. Chem. Soc.* **124**, 3238–44.
Wang, X., Parlow, J. J. & Porco, J. A. Jr. (2000). *Org. Lett.* **2**, 3509–3512.
Weber, E., Finge, S. & Csoregh, I. (1991). *J. Org. Chem.* **56**, 7281–7288.
Weber, E., Reutel, C., Foces-Foces, C. & Llamas-Saiz, A. L. (1994). *J. Chem. Soc. Perkin Trans.* 2, pp. 1455–1461.
Weber, E., Reutel, C., Foces-Foces, C. & Llamas-Saiz, A. L. (1995). *J. Phys. Org. Chem.* **8**, 159–170.