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2-Ethyl-4-hydroxy-3a,4,9,9a-tetrahydro-4,9-[1′,2′]benzeno-1*H*-benz[*f*]isoindole-1,3(2*H*)-dione

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Two molecules of the title compound, $C_{20}H_{17}NO_3$, are linked by a pair of hydroxy–amide interactions over a center of inversion, giving a hydrogen-bonded dimeric structure.

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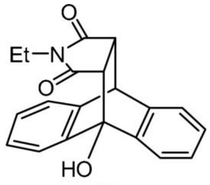
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.042
wR factor = 0.118
Data-to-parameter ratio = 11.8

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

Comment

Anthrone and its derivatives undergo a Diels–Alder reaction with dienophiles, and if the reaction is catalysed by a chiral catalyst, the product is an optically active cyclic compound (Shen *et al.*, 2006). For example, anthrone itself reacts with *N*-methylmaleimide to give the cycloadduct in high yield if a chiral β-aminoalcohol is used (Riant & Kagan, 1989); the corresponding cycloadduct is obtained for *N*-phenylmaleimide with a chiral pyrrolidone catalyst (Uemae *et al.*, 2001). In the present study, the use of triethylamine has yielded the racemic title cycloadduct, (I). The compound features a hydroxy group that interacts with one of the two amido O atoms, giving rise to a hydrogen-bonded dimer.



(I)

Experimental

Triethylamine (10 mg, 1 mmol) was added to a solution of anthrone (10 mg, 0.5 mmol) and *N*-ethylmaleimide (6.5 mg, 0.5 mmol) in

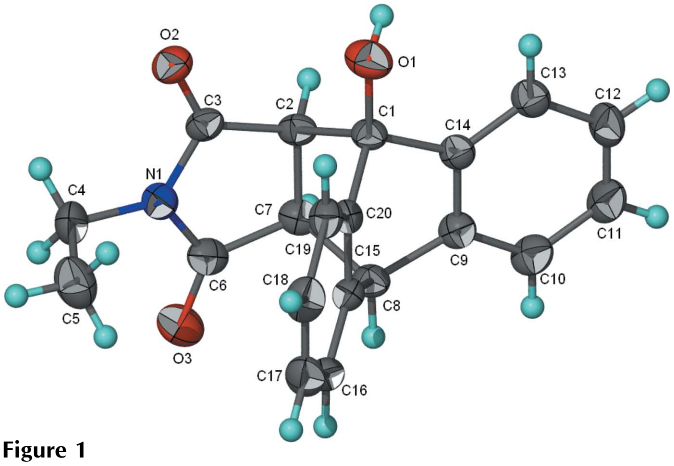


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level and H atoms as spheres of arbitrary radius. The minor disordered OH component is not shown.

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chloroform (5 ml). After the mixture was stirred to give a clear solution, 1 *M* HCl (5 ml) was added. The organic phase was separated and washed with saturated sodium chloride solution. The chloform solution was dried over magnesium sulfate. The solvent was removed and the residue purified by column chromatography on silica gel. Colorless crystals were obtained by recrystallization from ethanol.

Crystal data

$C_{20}H_{17}NO_3$
$M_r = 319.35$
Triclinic, $P\bar{1}$
$a = 7.743$ (2) Å
$b = 8.133$ (2) Å
$c = 13.718$ (4) Å
$\alpha = 74.57$ (2)°
$\beta = 85.06$ (2)°
$\gamma = 68.06$ (2)°
$V = 772.3$ (4) Å^3
$Z = 2$
$D_x = 1.373$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 295$ (2) K
Block, colorless
0.27 × 0.25 × 0.22 mm

Data collection

Enraf–Nonius CAD-4 four-circle diffractometer
ω–2θ scans
Absorption correction: none
2710 measured reflections
2707 independent reflections
1756 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.007$
$\theta_{\rm max} = 25.0°$
3 standard reflections every 300 reflections intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.118$
$S = 1.05$
2707 reflections
229 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0656P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.22$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.17$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*o*···O2$^{\rm i}$	0.82	1.97	2.778 (2)	169

Symmetry code: (i) $-x+1, -y+1, -z+1$.

The hydroxy group is disordered over two positions (at C1 and C8) with site-occupation factors of 0.847 (4) and 0.153 (4). The C—O distances of the two components were restrained to be equal. C-bound H atoms were placed at calculated positions (C—H = 0.93–0.98 Å) and were included in the refinement in the riding-model approximation, with $U_{\rm iso}$(H) set at 1.2 $U_{\rm eq}$(C) or 1.5 times $U_{\rm eq}$(methyl C). The hydroxy H atoms were also placed at calculated positions (O—H = 0.82 Å) and $U_{\rm iso}$(H) set at $1.5U_{\rm eq}$(O). The torsion angles about the C—O bonds were also refined. Although the occupancy of the minor component is only 0.153 (4), its omission gave a large peak (1.13 e Å^{-3}) in the final difference Fourier map.

Data collection: *DIFRAC* (Gabe *et al.*, 1993); cell refinement: *DIFRAC*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2006).

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