cascade least-squares refinement, H atoms located in difference maps and refined with isotropic thermal parameters; $R = 0.0571$, $wR = 0.0386$ for 206 parameters and 2221 reflections ($R = 0.0846$ and $wR = 0.0404$ for all 3095 unique reflections), $S = 1.801$, $(\Delta/\sigma)_{max} = 0.004$; largest peaks in the final difference map of -0.70 and $0.90\,e\,\text{Å}^{-3}$ (adjacent to Br atoms); $\Sigma w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o) + 0.00002F_o^2]^{-1}$; isotropic extinction correction applied, $F = F_c/[1.0 + 2.6\,(4) \times 10^{-6}F_c^2/\sin(2\theta)]^{0.25}$. All computer programs supplied by Nicolet Instrument Corporation (1986) for Desktop 30 Microeclipse and Nova 4/C configuration; atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV). Fig. 1 is a drawing of compound (3), Table 1 lists atomic positional parameters while Table 2 gives interatomic distances and bond angles.*

Related literature. The synthesis and reactions of brendanes (Arbuzov, Gevorkyan, Pekhk, Bobyleva & Belikova, 1984; Adam, Carballeira, Peters, Peters & Von Schnering, 1983; Hamlin & Toyne, 1981; Casanova, Waegell, Koukoua & Toure, 1979; Nickon, Kwasnik, Mathew, Swartz, Williams & DiGiorgio, 1978) and oxabrendanes (Inokuma, Sugie, Moriguchi, Shimomura & Katsube, 1982; Ranganathan, Ranganathan & Mehrotra, 1976;

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* Lists of H-atom coordinates, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53410 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Ethyl 4-Oxo-1,2,3,4-tetrahydrocinnoline-2-acetate

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Abstract. $C_{12}H_{14}N_2O_3$, $M_r = 234.3$, monoclinic, $P2_1/c$, $a = 8.760\,(1)$, $b = 13.550\,(3)$, $c = 10.456\,(2)$ Å, $\beta = 104.62\,(1)°$, $V = 1200.9\,(4)$ Å^3, $Z = 4$, $D_x = 1.296$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, Ni filter, $\mu = 0.74$ mm^{-1}, $F(000) = 496$, $T = 293$ K. Final $R = 0.062$ for 2182 observed reflections. 4-Oxo-1,2,3,4-tetrahydrocinnoline is in its standard N(2) sofa conformation. The orientation of the ethyl acetate group

Table 1. *Final atomic coordinates and equivalent isotropic thermal parameters for non-*H *atoms*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i{}^*a_j{}^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq} (Å²)
O(4)	0·1613 (2)	0·0630 (2)	0·3871 (2)	6·41 (5)
O(12)	0·3157 (2)	−0·0051 (1)	0·9830 (1)	4·96 (4)
O(13)	0·1235 (2)	0·1077 (1)	0·9423 (1)	4·57 (3)
N(1)	0·4491 (2)	0·0042 (1)	0·7441 (2)	3·83 (4)
N(2)	0·2904 (2)	−0·0296 (1)	0·7146 (1)	3·65 (3)
C(3)	0·2402 (2)	−0·0458 (1)	0·5718 (2)	4·29 (5)
C(4)	0·2525 (2)	0·0462 (1)	0·4931 (2)	4·16 (4)
C(5)	0·4126 (3)	0·1987 (1)	0·4906 (2)	4·98 (5)
C(6)	0·5378 (3)	0·2577 (1)	0·5487 (3)	5·86 (7)
C(7)	0·6379 (3)	0·2307 (2)	0·6698 (3)	5·89 (7)
C(8)	0·6134 (2)	0·1458 (1)	0·7339 (2)	4·69 (5)
C(9)	0·4810 (2)	0·0871 (1)	0·6783 (2)	3·52 (4)
C(10)	0·3829 (2)	0·1123 (1)	0·5552 (2)	3·76 (4)
C(11)	0·1868 (2)	0·0400 (1)	0·7584 (2)	3·69 (4)
C(12)	0·2185 (2)	0·0432 (1)	0·9069 (2)	3·54 (4)
C(14)	0·1401 (2)	0·1202 (2)	1·0836 (2)	4·93 (6)
C(15)	0·0229 (3)	0·1956 (2)	1·0986 (3)	6·44 (8)

Table 2. *Selected bond lengths* (Å), *angles* (°) *and torsional angles* (°) *with e.s.d.'s in parentheses*

N(1)—N(2)	1·422 (2)	C(4)—C(10)	1·468 (2)
N(1)—C(9)	1·382 (2)	C(8)—C(9)	1·406 (2)
N(2)—C(3)	1·463 (2)	C(9)—C(10)	1·398 (3)
C(3)—C(4)	1·513 (2)	N(2)—C(11)	1·460 (2)
C(4)—O(4)	1·213 (3)		

N(2)—N(1)—C(9)	117·6 (2)	C(3)—C(4)—C(10)	114·6 (2)
N(1)—N(2)—C(3)	107·2 (2)	C(3)—C(4)—O(4)	122·1 (2)
N(1)—N(2)—C(11)	112·0 (1)	O(4)—C(4)—C(10)	123·3 (2)
C(3)—N(2)—C(11)	111·5 (1)	C(4)—C(10)—C(9)	119·2 (1)
N(2)—C(3)—C(4)	112·8 (1)	N(1)—C(9)—C(10)	120·2 (2)

C(9)—N(1)—N(2)—C(3)	55·7 (7)	C(4)—C(10)—C(9)—N(1)	−4·1 (3)
N(1)—N(2)—C(3)—C(4)	−58·4 (2)	C(10)—C(9)—N(1)—N(2)	−25·1 (2)
N(2)—C(3)—C(4)—C(10)	32·8 (2)	H(1)—N(1)—C(9)—C(8)	11 (2)
C(3)—C(4)—C(10)—C(9)	−0·8 (2)	H(1)—N(1)—N(2)—C(11)	80 (2)

is determined by an intramolecular hydrogen bond between the carbonyl oxygen and N(1) with an N···O distance of 3·016 (3) Å.

Experimental. Prismatic crystals of the compound were crystallized from ethanol. Accurate cell dimensions were calculated from 25 reflections (θ range 20–28°) and data collection was from the same crystal, of dimensions 0·34 × 0·21 × 0·16 mm, on a CAD-4 diffractometer. Of the 2024 independent reflections measured in the $\omega/2\theta$ scan mode (h_{max}, k_{max}, ±l_{max} = 10, 16, 13) to $\theta_{max} = 75°$, 2182 with $I > 2\sigma(I)$ were considered observed and used in the refinement. Three standards monitored during data collection ($\overline{4}14$, $\overline{2}44$ and 035) showed variations in intensity of less than 2 %. The data were corrected for Lorentz and polarization effects. Absorption was neglected. Structure was solved by direct methods (*SHELXS*86; Sheldrick, 1986) and refined by full-matrix least squares (*SHELX*76; Sheldrick, 1976) with anisotropic temperature factors for non-H atoms and isotropic factors for H atoms found on

ΔF maps. The function minimized was $\Sigma w(|F_o| - |F_c|)^2$, where $w^{-1} = \sigma^2(F_o) + 0.0099F_o^2$. The refinement converged to a maximum shift/e.s.d. = 0·02 (scale factor). $R = 0.062$, $wR = 0.075$ and $S = 0.941$. The maximum and minimum peaks in the final Fourier synthesis were 0·22 and −0·20 e Å⁻³, respectively. All calculations were performed on an Amstrad PC 1512 microcomputer. Scattering factors were those incorporated in *SHELX*76.

A general view of the molecule and atomic labeling scheme are shown in Fig. 1, and the final atomic coordinates for the non-H atoms are shown in Table 1.* Bond lengths and angles are given in Table 2.

Related literature. The 4-oxo-1,2,3,4-tetrahydro-cinnoline system has been utilized as a framework for new proposed non-narcotic analgesics (Stańczak, 1987; Groszkowski & Stańczak, 1989; Kwapiszewski, Stańczak & Groszkowski, 1990). The first report of the crystal structure of the system showed the geometry and electronic structure of the C(9)–N(1)–N(2) moiety to be dependent mostly on N(2) substitution (Główka & Iwanicka, 1991). The ester side chain is in an extended conformation stabilized by an intramolecular hydrogen bond N(1)—H···O(12) [N···O 3·016 (3) Å, N—H···O 111 (2)°]. Two other crystal structures containing the 1,2,3,4-tetrahydrocinnoline framework are known (Lai, Mallakpour, Butler & Palenik, 1985; Bieri, Meier & Heimgartner, 1978), but the conformation of the 1,2-diazine ring in these structures is different due to its incorporation into a condensed three-ring system and the absence of the 4-oxo function.
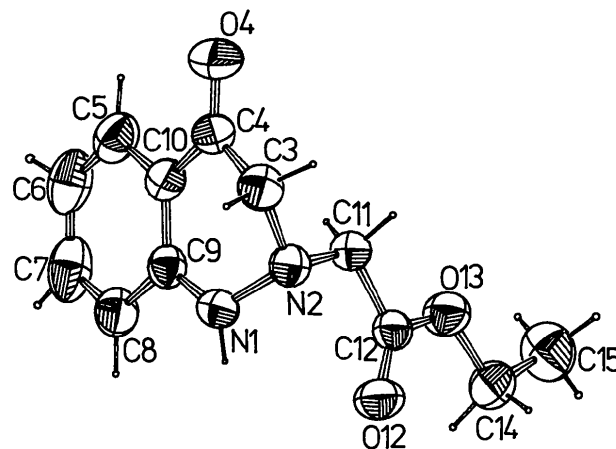
Fig. 1. Overall view of the molecule and the atomic labelling scheme (Johnson, 1976).

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Structure of 10,11-Dihydro-5,10-ethano-5H-dibenzo[a,d]cycloheptan-11-one

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Abstract. $C_{17}H_{14}O$ (1), $M_r = 234.3$, monoclinic, $P2_1/c$, $a = 9.587$ (2), $b = 11.091$ (2), $c = 11.815$ (2) Å, $\beta = 102.00$ (1)°, $V = 1228.8$ (4) Å^3, $Z = 4$, $D_x = 1.266$ g cm^{-3}, Mo $K\alpha$ ($\lambda = 0.71073$ Å), $\mu = 0.72$ cm^{-1}, $F(000) = 496$, $T = 193$ K, $R = 0.0387$ and $wR = 0.0411$ for 1529 reflections $[I \geq 3\sigma(I)]$. The molecule consists of two benzene rings fused on opposite sides of a cycloheptane ring with an ethano group bridging C(5) and C(10). The four-membered chain of C(5), C(13), C(12) and C(10) is nearly planar [angle C(5)—C(13)—C(12)—C(10) is 6.8 (3)°], and it forms dihedral angles of 118.0 (4) and 128.9 (4)° with the C(1) and the C(6) phenyl rings, respectively. The dihedral angle between the phenyl rings is 112.9 (4)°. The two 7-membered rings [C(11a), C(4a), C(5), C(13), C(12), C(10), C(11) and C(11a), C(4a), C(5), C(5a), C(9a), C(10). C(11)] are in boat–sofa conformations, BS11a and BS11a, respectively. Their puckering parameters [Cremer & Pople (1975). J. Am. Chem. Soc. 97, 1354–1358] q(2), q(3), φ(2), φ(3) and the total puckering amplitude, Q, are 0.943, 0.356 Å, 185.2, 2.3°, 1.008 Å, for the former, and 0.925, 0.301 Å, 355.9, 182.0°, 0.973 Å, for the latter. The 6-membered ring [C(5), C(13), C(12), C(10), C(9a), C(5a)] is in a boat conformation, ^{5,10}B, and it's puckering parameters q(2), q(3), φ(2), and the puckering amplitude, Q, are 0.688, 0.022 Å, 175.4°, 0.689 Å, respectively. The carbonyl oxygen is involved in two close intermolecular contacts with the aromatic H(7) and H(9) atoms [O(1)···H(7), ($-x$, $0.5 + y$, $1.5 - z$) = 2.53 (2) Å,

O(1)···C(7) = 3.36 (1) Å, O(1)···H(7)—C(7) = 117 (1)°; O(1)···H(9), ($-x$, $1 - y$, $1 - z$) = 2.62 (2) Å, O(1)···C(9) = 3.61 (1) Å, O(1)···H(9)—C(9) = 141 (1)°].

Experimental. A 50 ml round-bottom flask equipped with a reflux condenser and a magnetic stirring bar was charged with 973 mg (4.1 mmol) of 10,11-dihydro-5,10-ethano-5H-dibenzo[a,d]cyclohepten-11-ol, 788 mg (4.4 mmol) of N-bromosuccinimide, 12 mg of benzoyl peroxide and 20 ml of carbon tetrachloride. The mixture was stirred and irradiated with a 150 W unfrosted incandescent bulb until the N-bromosuccinimide was completely consumed (about 45 min). The mixture was washed with two 10 ml portions of water, one 10 ml portion of saturated sodium hydrogencarbonate solution, and two 10 ml portions of 0.05 M sodium thiosulfate solution. The organic layer was stripped of solvent and the residue dissolved in chloroform; that solution was eluted through a short column of activated alumina. The eluate was concentrated, affording a bright-yellow oil that crystallized on standing to yield 855 mg (90% of theoretical) of 10,11-dihydro-5,10-ethano-5H-dibenzo[a,d]cyclohepten-11-one, m.p. 383–386 K. The product was identified by ^{1}HMR and by ^{13}CMR. The crystal used in the X-ray analysis was selected from a few excellent specimens produced in a single crystallization from a solution of the product in a 10:1 mixture of hexanes and chloroform, and was a pale-yellow prism of dimen-