Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* ($Å^2$) *for compound* (II)

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

	x	y	z	U_{eq}
P	0.57852 (5)	0.79756 (4)	0.31188 (2)	0.02374 (11)
O1	0.5135 (2)	0.97347 (12)	0.34408 (6)	0.0293 (2)
O2	0.4068 (2)	0.75279 (15)	0.24391 (6)	0.0325 (2)
O3	0.7976 (2)	0.78828 (14)	0.28002 (7)	0.0362 (2)
O4	0.4832 (2)	0.8200 (2)	0.53022 (7)	0.0415 (3)
O5	0.1346 (2)	0.7934 (3)	0.52529 (11)	0.0705 (5)
C1	0.3062 (3)	0.7584 (2)	0.49913 (9)	0.0339 (3)
C2	0.3369 (2)	0.6411 (2)	0.42869 (8)	0.0298 (3)
C3	0.1735 (3)	0.5479 (3)	0.40176 (11)	0.0482 (4)
C4	0.5499 (2)	0.6362 (2)	0.39025 (8)	0.0291 (3)
N	0.9380 (2)	0.5515 (2)	0.16453 (9)	0.0364 (3)

Table 4. *Selected geometric parameters (Å, °) for compound* (II)

P—O1	1.517 (2)	O5—C1	1.209 (2)
P—O2	1.576 (2)	C1—C2	1.486 (2)
P—O3	1.496 (2)	C2—C3	1.325 (2)
P—C4	1.804 (2)	C2—C4	1.504 (2)
O4—C1	1.307 (2)		
O1—P—O2	104.8 (1)	O5—C1—O4	123.2 (2)
O1—P—O3	115.3 (1)	O5—C1—C2	123.4 (2)
O2—P—O3	111.6 (1)	O4—C1—C2	113.4 (2)
O1—P—C4	109.5 (1)	C3—C2—C1	118.3 (2)
O2—P—C4	106.0 (1)	C3—C2—C4	123.0 (2)
O3—P—C4	109.2 (1)	C1—C2—C4	118.7 (2)
C2—C4—P	113.0 (2)		
O1—P—C4—C2	50.3 (2)	O5—C1—C2—C3	−11.6 (3)
O2—P—C4—C2	−62.2 (2)	O4—C1—C2—C3	168.2 (2)
O3—P—C4—C2	177.4 (2)	O5—C1—C2—C4	167.2 (2)
C3—C2—C4—P	91.0 (2)	O4—C1—C2—C4	−13.0 (2)
C1—C2—C4—P	−87.7 (2)		

Table 5. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
Compound (I)				
O(11)—H(11)···O(1)	0.94 (5)	1.55 (5)	2.470 (3)	167 (5)
O(12)—H(12)···O(23)	0.91 (4)	1.60 (4)	2.511 (3)	176 (4)
O(15)—H(15)···O(24^{i})	0.84 (4)	1.83 (4)	2.661 (3)	172 (4)
O(21)—H(21)···O(13^{ii})	0.78 (4)	1.79 (4)	2.572 (3)	179 (4)
O(22)—H(22)···O(13^{iii})	0.83 (5)	1.76 (5)	2.578 (3)	164 (4)
O(25)—H(25)···O(14^{iv})	0.97 (5)	1.70 (4)	2.657 (3)	169 (4)
O(1)—H(1)···O(12^{v})	0.83 (4)	1.98 (4)	2.813 (4)	173 (4)
O(1)—H(2)···O(23^{vi})	0.82 (5)	1.98 (4)	2.790 (4)	166 (4)
Compound (II)				
O(2)—H(2)···O(1^{vii})	0.71 (3)	1.94 (3)	2.650 (3)	176 (3)
O(4)—H(4)···O(1^{viii})	0.75 (4)	1.88 (4)	2.609 (3)	165 (4)
N—H(11)···O(5^{ix})	0.89 (3)	2.00 (3)	2.893 (2)	176 (3)
N—H(12)···O(3^{x})	0.89 (3)	1.88 (3)	2.768 (3)	174 (3)
N—H(13)···O(3)	0.94 (3)	1.90 (3)	2.796 (3)	159 (3)
N—H(14)···O(1^{vii})	0.89 (3)	2.07 (3)	2.918 (3)	160 (3)

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

Data collection: *XTL/XTLE* system (Syntex, 1976) for (I); KM-4 software (Kuma, 1989) for (II). Cell refinement: *XTL/XTLE* system for (I); KM-4 software for (II). Data reduction: *XTL/XTLE* system for (I); KM-4 software for (II). For both compounds, program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1185). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3,4-Dihydro-3-methyl-6-nitro-2*H*-1,3-benzoxazin-2-one and *p*-Nitrophenyl Dimethylcarbamate

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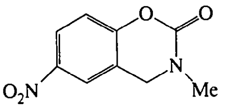
Abstract

In 3,4-dihydro-3-methyl-6-nitro-2*H*-1,3-benzoxazin-2-one ($C_9H_8N_2O_4$) (1), the plane of the *cis* carbamate group is 11.1 (2)° to that of the benzene ring, whereas, in the acyclic analogue, *p*-nitrophenyl dimethylcarbamate ($C_9H_{10}N_2O_4$) (2), the corresponding angle is 51.88 (9)° and the configuration is *transoid*. In (1), the O1—C7—O2 angle is 115.4 (3)° and the O1—C7—N1 angle is 117.3 (2)°; in (2), the corresponding angles are 122.8 (2) and 110.8 (2)°, respectively.

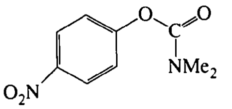
Comment

The title compounds have been used as modifiers of enzymes such as chymotrypsin (Kitson & Freeman,

1993) and aldehyde dehydrogenase (Kitson & Kitson, 1994). The cyclic carbamate (1) provides a useful 'reporter' group (Burr & Koshland, 1964) for probing an enzyme's active site and has also been valuable in the preparation of crystals of aldehyde dehydrogenase suitable for X-ray structural analysis (Baker *et al.*, 1994). We have observed that (1), but not (2), is surprisingly light-sensitive in aqueous solution (Kitson & Freeman, 1993).

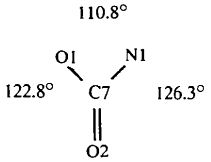


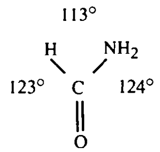
(1)



(2)

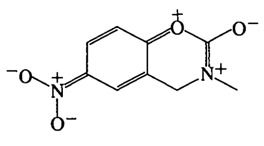
Figs. 1 and 2 show the structures of (1) and (2) and the numbering of the atoms. The major differences in the two structures are to be found in the angles around the carbonyl group; there are no very significant differences in bond lengths. In (2), the unconstrained carbamate group has angles similar to those found in a simple amide such as methanamide (Kagan, 1979); the comparison is shown below.





The bulky aromatic ring and —$N(CH_3)_2$ groups adopt an approximately *trans* configuration with respect to the O1—C7 bond, but the plane of the carbamate group lies at 51.88 (9)° to the plane of the benzene ring, presumably to avoid interaction between the orbitals of O2 and the H atom on C2.

In (1), the maximum amount of resonance stabilization exists if the second ring is coplanar with the benzene ring, as shown below. However, the methylene group at C8 evidently precludes this from occurring. The angle C2—C8—N1 has a value of 111.1 (2)° (typical of bonds involving methylene groups), and not close to 120° as demanded by the structure below.



The presence of this CH_2 group is clearly the factor that causes the plane of the (necessarily *cis*) carbamate group to be tilted 11.1 (2)° with respect to the benzene ring. However, the O1—C7—N1 angle is perturbed closer to 120° than in the 'free' carbamate (2) [the angles are 117.3 (2) and 110.8 (2)°, respectively], at the expense of a similarly sized narrowing of the O1—C7—O2 angle [122.8 (2)° in (2) and 115.4 (3)° in (1)]. If the 6.5° difference in the O1—C7—N1 angle observed in the two compounds represents a significant degree of strain in the ring of (1), then this may explain why (1) is light-sensitive and (2) is not.

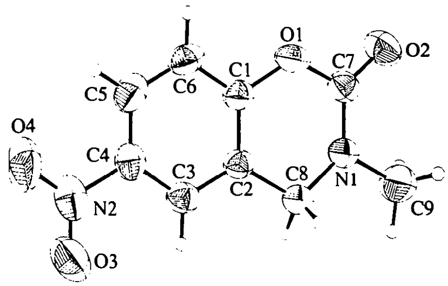


Fig. 1. Displacement ellipsoid diagram of (1) at the 50% probability level (arbitrary levels for H atoms) showing the numbering system used. H atoms are numbered according to the C atoms to which they are attached.

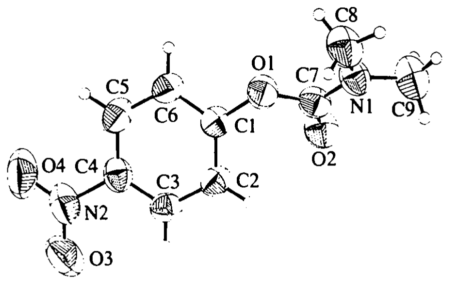


Fig. 2. Displacement ellipsoid diagram of (2) at the 50% probability level (arbitrary levels for H atoms) showing the numbering system used. H atoms are numbered according to the C atoms to which they are attached.

Aldehyde dehydrogenase reacts significantly faster with (1) than with (2), in both the presence and the absence of NAD^+ (Kitson & Kitson, 1994). This difference will presumably become explicable when the three-dimensional structure of the enzyme is elucidated (Baker *et al.*, 1994) and the architecture of the active site can then be related to the structure reported here.

Experimental

Compounds (1) and (2) were prepared as previously reported (Kitson, 1989; Kitson & Freeman, 1993) and recrystallized from alcohol.

Compound (1)

Crystal data

$C_9H_8N_2O_4$	Mo $K\alpha$ radiation
$M_r = 208.17$	$\lambda = 0.71073$ Å

Monoclinic
$P2_1/n$
$a = 6.042\,(2)$ Å
$b = 13.688\,(3)$ Å
$c = 10.904\,(3)$ Å
$\beta = 99.13\,(3)°$
$V = 890.4\,(4)$ Å^3
$Z = 4$
$D_x = 1.553$ Mg m^{-3}

Cell parameters from 22 reflections
$\theta = 8.996$–$15.181°$
$\mu = 0.125$ mm^{-1}
$T = 293\,(2)$ K
Needle-like fragment
$0.45 \times 0.11 \times 0.09$ mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1713 measured reflections
1558 independent reflections
887 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.0324$

$\theta_{\max} = 24.96°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 16$
$l = -12 \rightarrow 12$
3 standard reflections monitored every 100 reflections frequency: 120 min intensity decay: 0.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0421$
$wR(F^2) = 0.1395$
$S = 1.227$
1558 reflections
137 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.1000P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.004$
$\Delta\rho_{\max} = 0.289$ e Å^{-3}
$\Delta\rho_{\min} = -0.221$ e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.006 (6)
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) for (1)*

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

	x	y	z	$U_{\rm eq}$
O1	0.1808 (3)	0.88314 (15)	−0.0110 (2)	0.0550 (6)
O2	0.1950 (4)	0.7902 (2)	0.1532 (2)	0.0632 (7)
O3	0.7480 (5)	1.0376 (2)	−0.4017 (2)	0.0865 (9)
O4	0.4800 (5)	1.1432 (2)	−0.4219 (3)	0.0954 (9)
N1	0.4784 (4)	0.7771 (2)	0.0407 (2)	0.0462 (6)
N2	0.5738 (5)	1.0700 (2)	−0.3746 (2)	0.0639 (8)
C1	0.2856 (4)	0.9274 (2)	−0.1004 (2)	0.0423 (7)
C2	0.4805 (4)	0.8899 (2)	−0.1319 (2)	0.0384 (7)
C3	0.5747 (4)	0.9373 (2)	−0.2230 (2)	0.0449 (7)
C4	0.4712 (5)	1.0191 (2)	−0.2787 (3)	0.0470 (7)
C5	0.2761 (5)	1.0557 (2)	−0.2472 (3)	0.0570 (9)
C6	0.1811 (5)	1.0090 (2)	−0.1567 (3)	0.0524 (8)
C7	0.2874 (5)	0.8123 (2)	0.0672 (3)	0.0454 (7)
C8	0.5810 (5)	0.7999 (2)	−0.0684 (3)	0.0489 (8)
C9	0.5890 (6)	0.7005 (3)	0.1199 (3)	0.0732 (11)

Table 2. *Selected geometric parameters (Å, °) for (1)*

O1—C7	1.381 (3)	N2—C4	1.473 (4)
O1—C1	1.383 (3)	C1—C6	1.379 (4)
O2—C7	1.204 (3)	C1—C2	1.378 (3)
O3—N2	1.221 (3)	C2—C3	1.382 (4)
O4—N2	1.224 (3)	C2—C8	1.494 (4)
N1—C7	1.324 (4)	C3—C4	1.377 (4)
N1—C9	1.452 (4)	C4—C5	1.374 (4)
N1—C8	1.460 (3)	C5—C6	1.375 (4)
C7—O1—C1	121.4 (2)	C1—C2—C8	119.5 (2)
C7—N1—C9	117.9 (2)	C2—C3—C4	119.3 (3)
C7—N1—C8	126.7 (2)	C5—C4—C3	122.3 (3)
C9—N1—C8	115.1 (2)	C5—C4—N2	118.7 (3)
O3—N2—O4	123.7 (3)	C3—C4—N2	119.0 (3)
O3—N2—C4	118.4 (3)	C6—C5—C4	118.9 (3)
O4—N2—C4	117.9 (3)	C5—C6—C1	118.8 (3)
C6—C1—O1	116.1 (2)	O2—C7—N1	127.3 (3)
C6—C1—C2	122.7 (3)	O2—C7—O1	115.4 (3)
O1—C1—C2	121.2 (2)	N1—C7—O1	117.3 (2)
C3—C2—C1	118.1 (3)	N1—C8—C2	111.1 (2)
C3—C2—C8	122.5 (2)		

Compound (2)

Crystal data

$C_9H_{10}N_2O_4$
$M_r = 210.19$
Orthorhombic
$Pca2_1$
$a = 11.207\,(2)$ Å
$b = 12.1950\,(10)$ Å
$c = 7.3631\,(9)$ Å
$V = 1006.3\,(2)$ Å^3
$Z = 4$
$D_x = 1.387$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 24 reflections
$\theta = 11.665$–$16.343°$
$\mu = 0.111$ mm^{-1}
$T = 293\,(2)$ K
Hexagonal plate
$0.55 \times 0.38 \times 0.22$ mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2529 measured reflections
1567 independent reflections
990 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.0117$

$\theta_{\max} = 29.96°$
$h = 0 \rightarrow 15$
$k = -14 \rightarrow 17$
$l = 0 \rightarrow 10$
3 standard reflections monitored every 100 reflections frequency: 120 min intensity decay: 0.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0465$
$wR(F^2) = 0.1433$
$S = 1.240$
1567 reflections
137 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.1000P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.008$
$\Delta\rho_{\max} = 0.183$ e Å^{-3}
$\Delta\rho_{\min} = -0.192$ e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.023 (6)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)
Flack parameter = −1.6 (21)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (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	$U_{\rm eq}$
O1	0.0693 (2)	0.34979 (14)	0.3098 (3)	0.0631 (6)
O2	0.2431 (3)	0.3098 (2)	0.4537 (4)	0.0689 (6)
O3	0.2260 (3)	0.8285 (2)	0.1249 (6)	0.1099 (13)
O4	0.0548 (3)	0.8586 (2)	0.2501 (7)	0.1106 (12)
N1	0.1191 (2)	0.1755 (2)	0.3557 (4)	0.0636 (7)
N2	0.1359 (3)	0.7973 (2)	0.2030 (5)	0.0753 (8)

C1	0.0953 (2)	0.4610 (2)	0.2955 (4)	0.0493 (6)
C2	0.2009 (2)	0.4990 (2)	0.2218 (4)	0.0531 (6)
C3	0.2141 (2)	0.6101 (2)	0.1942 (4)	0.0548 (7)
C4	0.1227 (2)	0.6799 (2)	0.2399 (4)	0.0565 (7)
C5	0.0183 (2)	0.6428 (2)	0.3166 (5)	0.0622 (8)
C6	0.0054 (2)	0.5316 (2)	0.3461 (4)	0.0565 (7)
C7	0.1531 (2)	0.2788 (2)	0.3783 (4)	0.0555 (7)
C8	0.0117 (3)	0.1424 (3)	0.2588 (7)	0.0814 (11)
C9	0.1906 (4)	0.0890 (3)	0.4371 (7)	0.0845 (11)

Table 4. *Selected geometric parameters (Å, °) for (2)*

O1—C7	1.374 (3)	N2—C4	1.465 (4)
O1—C1	1.391 (3)	C1—C6	1.377 (4)
O2—C7	1.211 (4)	C1—C2	1.382 (4)
O3—N2	1.223 (5)	C2—C3	1.378 (4)
O4—N2	1.227 (4)	C3—C4	1.374 (4)
N1—C7	1.327 (4)	C4—C5	1.375 (4)
N1—C9	1.454 (5)	C5—C6	1.381 (4)
N1—C8	1.457 (4)		
C7—O1—C1	119.9 (2)	C3—C2—C1	118.6 (2)
C7—N1—C9	118.6 (3)	C4—C3—C2	119.5 (2)
C7—N1—C8	124.2 (3)	C3—C4—C5	122.1 (2)
C9—N1—C8	117.2 (3)	C3—C4—N2	119.0 (3)
O3—N2—O4	123.7 (3)	C5—C4—N2	118.9 (3)
O3—N2—C4	118.4 (3)	C4—C5—C6	118.4 (3)
O4—N2—C4	118.0 (3)	C1—C6—C5	119.7 (3)
C6—C1—C2	121.6 (3)	O2—C7—N1	126.3 (3)
C6—C1—O1	115.9 (2)	O2—C7—O1	122.8 (2)
C2—C1—O1	122.4 (2)	N1—C7—O1	110.8 (2)

For both (1) and (2), H atoms were placed in calculated sites riding on the C atoms to which they were attached. Isotropic displacement parameters were fixed at 0.08 Å^2.

For both compounds, data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ZORTEP* (Zsolnai, 1994); software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1035). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Pinacol Precursor to a Chiral Spiro Compound

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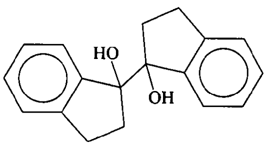
Abstract

The dimer of 1-indanolyl obtained from the UV photolysis of 1-indanone in 2-propanol is *trans*-α,α'-biindanyl-α,α'-diol, $(C_9H_9O)_2$. There are two almost identical molecules in the asymmetric unit linked by a hydrogen-bonding system that extends along the *a* axis. Bond distances and angles are as expected.

Comment

The diol dimer described here, (I), was prepared in the hope that intramolecular hydrogen bonding would lock it in the correct conformation for enantioselective rearrangement during pinacol rearrangement to produce a spiro compound with a chiral spiro atom. The molecule was the one expected, but all the hydrogen bonding is intermolecular, this despite IR studies on the compound in solution which indicated intramolecular hydrogen bonding (McBay & Abulū, 1994). In the crystal, the diol molecules form hydrogen-bonded pairs about centers of symmetry with O···O distances 2.716 (3) and 2.725 (3) Å; the O···H distances are 1.94 (3) and 1.99 (3) Å, with O—H···O angles of 152 (3) and 148 (3)°. These pairs are each bonded to



(I)

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