metry code: (i) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$] which links the molecules in quasi-planar chains along the y axis. Thus, *N*-acetylation simplifies the hydrogen-bond network found in 2-thiohydantoin which creates two-dimensional sheets rather than ribbons (Walker *et al.*, 1969).

Experimental

The title compound was prepared by refluxing 3.32 g of thiohydantoin in 20 ml of acetic anhydride for 30 min. The solid formed was filtered off and washed with ethyl ether. Crystals were obtained by slow evaporation of an acetone solution.

Crystal data

$C_5H_6N_2O_2S$
$M_r = 158.18$
Monoclinic
$P2_1/n$
$a = 8.2968\ (11)$ Å
$b = 7.7364\ (11)$ Å
$c = 10.6066\ (15)$ Å
$\beta = 93.434\ (11)°$
$V = 679.6\ (2)$ Å^3
$Z = 4$
$D_x = 1.546$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9.07–18.41°$
$\mu = 0.411$ mm^{-1}
$T = 293\ (2)$ K
Prism
0.20 × 0.15 × 0.15 mm
Colourless

Data collection

Enraf–Nonius MACH3 diffractometer
ω scans
Absorption correction: none
1458 measured reflections
1384 independent reflections
1026 reflections with $I > 2\sigma(I)$

$R_{int} = 0.031$
$\theta_{max} = 26.29°$
$h = -10 \rightarrow 10$
$k = -9 \rightarrow 0$
$l = 0 \rightarrow 13$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.115$
$S = 1.038$
1384 reflections
116 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.0632P)^2 + 0.2702P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.214$ e Å^{-3}
$\Delta\rho_{min} = -0.230$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.035 (5)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

S—C1	1.638 (2)	N1—C2	1.469 (3)
O3—C3	1.218 (3)	N2—C3	1.362 (3)
O4—C4	1.208 (3)	N2—C1	1.381 (3)
N1—C1	1.370 (3)	C2—C3	1.492 (3)
N1—C4	1.409 (3)	C4—C5	1.486 (4)
C1—N1—C4	131.4 (2)	N1—C2—C3	102.5 (2)
C1—N1—C2	111.3 (2)	O3—C3—N2	125.4 (2)
C4—N1—C2	117.4 (2)	O3—C3—C2	128.2 (2)
C3—N2—C1	113.6 (2)	N2—C3—C2	106.4 (2)
N1—C1—N2	106.1 (2)	O4—C4—N1	117.3 (2)
N1—C1—S	132.0 (2)	O4—C4—C5	122.3 (2)
N2—C1—S	121.9 (2)	N1—C4—C5	120.3 (2)

Data collection: *CAD-4 EXPRESS Software* (Enraf–Nonius, 1995). Cell refinement: *CAD-4 EXPRESS Software*. Data reduction: *HELENA* (Spek, 1996). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1996). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1500). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). C**54**, 428–430

(3*S*)-4,4-Dimethyl-2-oxotetrahydrofuran-3-yl (2*S*)-2-(1,4-Benzodioxin-6-yl)propionate

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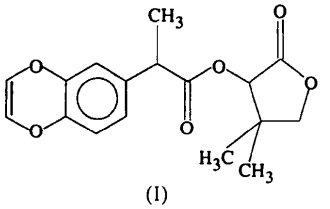
Abstract

The 2-oxofuran moiety in the title compound, $C_{17}H_{18}O_6$, has a skew-envelope form. The heterocycle of the 1,4-benzodioxin-6-yl moiety has the typical half-chair form and steric hindrance between the substituents produces

the observed loss of planarity of the 2-arylethanoic acid moiety.

Comment

The structure of the title compound, (I), a key intermediate in the synthesis of acidic anti-inflammatory agents, is reported. Among the non-steroidal anti-inflammatory agents, 2-arylpropionic acids constitute an important class. Although most of them are used in a racemic form, those having an *S* configuration at the propionate chiral centre are the most active (Rieu *et al.*, 1986; Sonawane *et al.*, 1992). The use of enantiomerically pure drugs in therapeutics is becoming of prime importance and as a result, much work has been performed in recent years to develop methods for the preparation of (*S*)-2-arylpropionic acids.



(I)

The 2-oxofurans usually have envelope forms, each with the C4 atom out of the plane defined by the remaining four atoms (Cobbledick & Small, 1987), but a 2-oxofuran with the 3 position substituted usually has an envelope form with the C3 atom deviating from the plane. The 2-oxofuran moiety of the title compound has a C3-envelope form, with a C4—O1—C1—C2 torsion angle of 4.8 (5)°. This form has also been observed in nepetaefolinol (Blount & Manchand, 1980), (*S*)-homoserinelactone hydrochloride (Papaioannou *et al.*, 1990), α-benzyloxy-γ-butyrolactone (Bocelli & Grenier-Loustalot, 1980), (4*R*,5*R*)-5-benzoyloxy-1,2-diaza-7-oxaspiro[4.4]non-1-en-6-one (Jonas *et al.*, 1991) and α-(1-thyminylmethyl)-γ-butyrolactone (Das *et al.*, 1993).

The moiety formed by atoms C2, O3, C7, O4 and C8 is nearly planar, with the C2—O3—C7—C8 torsion angle equal to 175.7 (4)°, which is explained by the steric hindrance between the O4 atom and the methyl substituents of the 2-oxofuran ring.

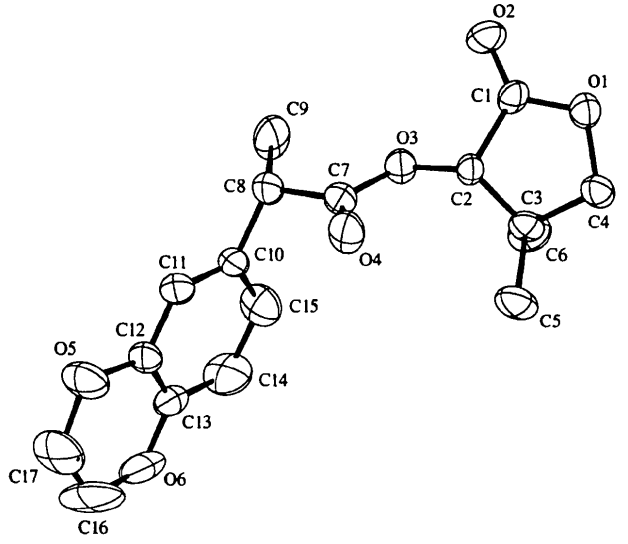


Fig. 1. Plot of one of the disordered conformations of the title molecule showing the numbering scheme and 50% probability displacement ellipsoids.

Experimental

The method followed for the asymmetric synthesis of α-arylpropionic acid from the corresponding racemic mixture involves the use of D-pantolactone and other homochiral alcohols (Larsen *et al.*, 1989; Calmes *et al.*, 1994). The separation of the diastereomeric mixture was achieved by flash chromatography and the diastereomeric purity was assessed by HPLC (diastereomeric excess > 99%). The two diastereomers were fully characterized by spectroscopy and elemental analysis.

Crystal data

$C_{17}H_{18}O_6$
M_r = 318.32
Orthorhombic
$P2_12_12_1$
a = 26.094 (4) Å
b = 9.717 (2) Å
c = 6.325 (2) Å
V = 1603.7 (7) Å^3
Z = 4
D_x = 1.318 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 12–21°
μ = 0.101 mm^{-1}
T = 293 (2) K
Prism
0.4 × 0.2 × 0.2 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
2683 measured reflections
2683 independent reflections
1022 reflections with $I > 2\sigma(I)$
θ_{max} = 29.98°
$h = 0 \rightarrow 36$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 8$
3 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.159
S = 0.949
2664 reflections
220 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0688P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.005
$\Delta\rho_{max}$ = 0.172 e Å^{-3}
$\Delta\rho_{min}$ = −0.168 e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.020 (4)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—C1	1.300 (5)	O4—C7	1.169 (5)
O1—C4	1.486 (5)	C1—C2	1.532 (5)
O2—C1	1.192 (4)	C2—C3	1.500 (6)
O3—C7	1.355 (5)	C3—C4	1.503 (5)
O3—C2	1.405 (4)	C7—C8	1.485 (6)
C1—O1—C4	109.6 (3)	C3—C2—C1	101.3 (3)
C7—O3—C2	118.8 (3)	C2—C3—C4	100.2 (3)
O2—C1—O1	124.3 (4)	O1—C4—C3	104.0 (3)
O2—C1—C2	126.6 (4)	O4—C7—O3	121.0 (4)
O1—C1—C2	109.1 (3)	O4—C7—C8	124.0 (5)
O3—C2—C3	118.4 (3)	O3—C7—C8	115.0 (4)
O3—C2—C1	109.6 (3)		

The C16 atom is disordered over two positions; the occupancy factor for each position was refined, resulting in a value of 0.83 (6) for C16 and 0.17 (6) for C16′. The positions of all H atoms were computed and refined with an overall isotropic displacement parameter, using a riding model. The enantiomer was defined according to the pharmacological activity of the compound.

Data collection: *CAD-4/PC* (Kretschmar, 1996). Cell refinement: *CAD-4/PC*. Data reduction: *CFEO* (Solans, 1978). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEP* (Brueggemann & Schmid, 1990). Software used to prepare material for publication: *PLATON* (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1022). Services for accessing these data are described at the back of the journal.

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Three *N*-Aryl-Substituted 3-Hydroxypyridin-4-ones

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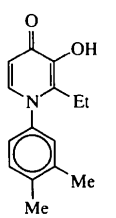
Abstract

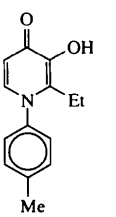
The molecular structures of 1-(3,4-dimethylphenyl)-2-ethyl-3-hydroxypyridin-4-one, $C_{15}H_{17}NO_2$, (1), 2-ethyl-3-hydroxy-1-(4-methylphenyl)pyridin-4-one, $C_{14}H_{15}NO_2$, (2), and 2-ethyl-3-hydroxy-1-(4-methoxyphenyl)-pyridin-4-one, $C_{14}H_{15}NO_3$, (3), have been determined. The compounds all exhibit mutually hydrogen-bonded dimeric pairs. In the case of (1), the molecules of the dimer are symmetry related, while in (2) and (3) two independent molecules of the asymmetric unit are linked.

Comment

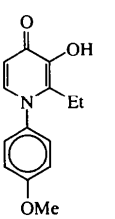
The structures form dimeric units through mutual O1—H1···O2 hydrogen bonds (Fig. 1). This type of dimeric structure is commonly found for the anhydrous 3-hydroxypyridin-4-ones (Hider *et al.*, 1990; Chan *et al.*, 1992; Xiao *et al.*, 1992; Burgess *et al.*, 1993) and 3-hydroxypyran-4-ones, which are also reported as hydrogen-bonded chains (Burgess *et al.*, 1996; Brown *et al.*, 1995).



(1)



(2)



(3)