

Table 4. Hydrogen-bonding geometry (Å, °) for (II)

D—H...A	D—H	H...A	D...A	D—H...A
N6—H61...O5 <sup>i</sup>	0.84	2.33	3.077 (3)	149
N6—H62...O3 <sup>ii</sup>	0.84	2.20	3.025 (3)	170
O3'—H3'O...N8	0.82	2.02	2.799 (3)	159
O5'—H5'O...N1 <sup>iii</sup>	0.82	2.08	2.896 (3)	176

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

All H atoms were found in difference Fourier syntheses but were constructed in geometrically reasonable positions and refined with a common isotropic displacement parameter. With the absence of suitable anomalous scatterers within the molecules, the determination of the absolute configuration was not possible from our X-ray data. However, comparison with the configuration of the parent molecules indicates that the proposed conformations are correct.

For both compounds, data collection: XSCANS (Siemens, 1996); cell refinement: XSCANS; data reduction: SHELXTL (Sheldrick, 1997b); program(s) used to solve structures: SHELXS97 (Sheldrick, 1990); program(s) used to refine structures: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1335). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1999). **C55**, 1950–1952

## 4-Phenyl-2,3,5,6,7,8-hexahydro-1H-pyrido[1,2-c]pyrimidine-1,3-dione

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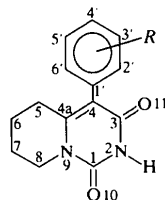
(Received 25 March 1999; accepted 1 July 1999)

## Abstract

In the structure of the title compound, C<sub>14</sub>H<sub>14</sub>N<sub>2</sub>O<sub>2</sub>, the saturated ring adopts a sofa conformation and the pyrimidine moiety is nearly planar. The mean planes of these fragments are close to coplanarity. The planar phenyl ring is twisted with respect to the pyrimidine-1,3-dione fragment. The molecules form centrosymmetric dimers *via* intermolecular N—H...O hydrogen bonds.

## Comment

The syntheses of the 4-aryl-hexahydro-1H-pyrido[1,2-c]pyrimidine-1,3-diones, (1)–(8), have been undertaken as a continuation of the search for new anxiolytic agents and studies on the relationship between structure and affinity to the 5-HT<sub>1A</sub> receptor for those compounds. The compounds designed are structurally related to buspirone, a drug widely used in the treatment of mental diseases (Goa & Ward, 1986; Taylor & Moon, 1991; Faludi, 1994). Buspirone shows affinity to 5-HT<sub>1A</sub>- and D<sub>2</sub>-receptor types and is functionally a partial agonist of the 5-HT<sub>1A</sub> receptors. Differences between the structures of buspirone and its new analogues are caused by a modification of the terminal imide moiety. It was noticed that as a result of this modification the lipophilicity of the molecule is higher and, consequently, the affinity to the 5-HT<sub>1A</sub> receptor increases (Raghupathi *et al.*, 1991; Lopez-Rodriguez *et al.*, 1996; Lopez-Rodriguez, Morcillo *et al.*, 1997; Lopez-Rodriguez, Rosado *et al.*, 1997).



- (1) R = H
- (2) R = 2'-Me
- (3) R = 3'-Me
- (4) R = 4'-Me
- (5) R = 4'-F
- (6) R = 4'-OMe
- (7) R = 2'-Cl
- (8) R = 4'-Cl

The present structural work has been undertaken to obtain more detailed information about the bond

system and conformation of 4-phenyl-2,3,5,6,7,8-hexahydro-1*H*-pyrido[1,2-*c*]pyrimidine-1,3-dione, (1), one of the substrates in the synthesis of new analogues of buspirone.

The molecular structure of (1), showing the labelling scheme, is presented in Fig. 1. Bond lengths and valency angles agree with standard values for analogous derivatives (Taylor & Kennard, 1982; Yamagata *et al.*, 1985; Lenstra *et al.*, 1991; Suresh *et al.*, 1996). Insignificant distortions of the saturated ring result from disorder of this fragment of the molecule.

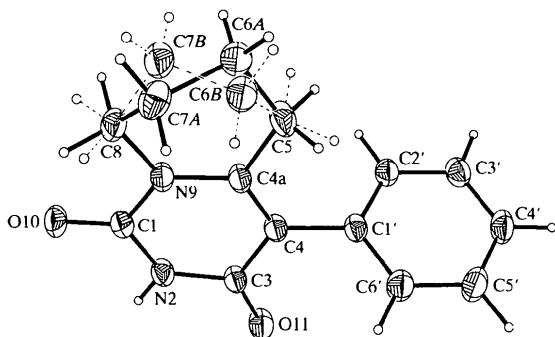


Fig. 1. A view of the title molecule showing the labelling scheme and disordered methylene groups. Displacement ellipsoids are drawn at the 30% probability level.

The C3=O11 bond length is insignificantly longer than C1=O10 (Table 1). The difference (*ca* 0.01 Å) could be induced by the intermolecular N2—H2...O11' hydrogen bond [N...O = 2.821 (2) Å; symmetry code: (i) 1 - *x*, 1 - *y*, 1 - *z*] *via* which centrosymmetric dimers are formed. The atoms within and around this hydrogen bond (O11, C3, N2, H2 and symmetry-related atoms) form an eight-membered ring which is almost flat [maximum deviation from the best plane is 0.037 (5) Å for O11]. Both carbonyl groups, C1=O10 and C3=O11, are involved in the short intermolecular C—H...O contacts, which are a feature of the crystal packing of the molecules. The geometric parameters of all intermolecular hydrogen bonds are listed in Table 2.

The C6 and C7 atoms in the saturated ring were found to be disordered. The occupancies of the alternative positions were refined to 0.493 (9) (C6A/C7A) and 0.507 (9) (C6B/C7B). Similar disorder was found for respective methylene-C atoms in (4) and (7) (Herold, Maciejewska & Wolska, 1999) and in a compound related to (1) (Knoch *et al.*, 1995). In both disorder components, the piperidine ring adopts a sofa conformation with C7A and C6B displaced by 0.74 (1) and 0.71 (1) Å, respectively, from the plane formed by the remaining five atoms. The conformation of this ring with either

C6A/C7A or C6B/C7B is slightly distorted from the typical sofa form, the asymmetry parameters according to Duax & Norton (1975) being  $\Delta C_s = 6.8$  (3) and  $\Delta C_s = 6.3$  (3)°, respectively.

This saturated ring is fused with the uracil moiety which is close to planarity [maximum deviations from the least-squares plane are 0.061 (1) Å for N9 and -0.060 (1) Å for C4]. The C5 and C8 atoms are above and C1' is below this plane by 0.027 (3), 0.252 (2) and -0.188 (2) Å, respectively. The best plane of the pyrimidine fragment is inclined to the mean piperidine planes formed by atoms C8/N9/C4a/C5/C6A and C8/N9/C4a/C5/C7B by only 5.1 (2) and 4.3 (2)°.

The disposition of the phenyl ring with respect to the pyrimidine-1,3-dione fragment can be described by the torsion angle C3—C4—C1'—C2' of 107.3 (2)°. The planar phenyl ring makes an angle of 72.24 (5)° with the plane of the above fragment. The aryl substituent at C4 cannot be coplanar with the pyrido[1,2-*c*]pyrimidine-1,3-dione system for steric reasons.

## Experimental

The synthesis of the title compound is described elsewhere (Herold, Wolska *et al.*, 1999). Crystals were grown from acetic acid by slow evaporation.

### Crystal data

C<sub>14</sub>H<sub>14</sub>N<sub>2</sub>O<sub>2</sub>  
*M<sub>r</sub>* = 242.27  
 Monoclinic  
*P*2<sub>1</sub>/*c*  
*a* = 11.101 (2) Å  
*b* = 8.404 (2) Å  
*c* = 12.836 (3) Å  
 $\beta$  = 92.68 (3)°  
*V* = 1196.2 (5) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.345 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

Cu *K*α radiation  
 $\lambda$  = 1.54178 Å  
 Cell parameters from 38 reflections  
 $\theta$  = 7.53–28.74°  
 $\mu$  = 0.743 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Prism  
 0.50 × 0.20 × 0.15 mm  
 Colourless

### Data collection

KM-4 four-circle diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction: none  
 2384 measured reflections  
 2259 independent reflections  
 1951 reflections with  
 $I > 2\sigma(I)$

*R*<sub>int</sub> = 0.010  
 $\theta_{\max}$  = 75.13°  
 $h = -13 \rightarrow 13$   
 $k = 0 \rightarrow 10$   
 $l = 0 \rightarrow 14$   
 3 standard reflections  
 every 100 reflections  
 intensity decay: 0.9%

### Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.042  
*wR*(*F*<sup>2</sup>) = 0.119

( $\Delta/\sigma$ )<sub>max</sub> = -0.009  
 $\Delta\rho_{\max}$  = 0.210 e Å<sup>-3</sup>  
 $\Delta\rho_{\min}$  = -0.173 e Å<sup>-3</sup>

$S = 1.102$   
 2256 reflections  
 207 parameters  
 H atoms treated by a  
 mixture of independent  
 and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0747P)^2 + 0.2332P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

Extinction correction:  
*SHELXL93* (Sheldrick,  
 1993)  
 Extinction coefficient:  
 0.0085 (10)  
 Scattering factors from  
*International Tables for  
 Crystallography* (Vol. C)

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Table 1. Selected geometric parameters (Å, °)

C1—O10	1.216 (2)	C3—O11	1.233 (2)
C1—N2	1.369 (2)	C4—C4a	1.359 (2)
N2—C3	1.379 (2)		
N2—C1—N9	115.44 (11)	C2'—C1'—C6'	118.72 (13)
N2—C3—C4	115.14 (11)		
N9—C4a—C5—C6B	30.7 (3)	C6A—C7A—C8—N9	56.9 (6)
N9—C4a—C5—C6A	−0.9 (5)	C6B—C7B—C8—N9	−39.0 (7)
C4a—C5—C6A—C7A	34.5 (8)	C5—C4a—N9—C8	−5.0 (2)
C5—C6A—C7A—C8	−62.3 (8)	C7A—C8—N9—C4a	−25.6 (4)
C4a—C5—C6B—C7B	−58.0 (6)	C7B—C8—N9—C4a	10.1 (4)
C5—C6B—C7B—C8	62.8 (8)		

Table 2. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
N2—H2...O11 <sup>i</sup>	0.90 (2)	1.92 (2)	2.821 (2)	175 (2)
C2'—H2'...O10 <sup>ii</sup>	0.97 (2)	2.55 (2)	3.472 (2)	159 (1)
C8—H8B...O11 <sup>iii</sup>	0.97	2.52	3.450 (2)	159
C8—H8C...O11 <sup>iii</sup>	0.97	2.55	3.450 (2)	154
C7A—H7C...O10 <sup>iv</sup>	0.97	2.81	3.430 (4)	122
C7B—H7C...O10 <sup>v</sup>	0.97	2.72	3.241 (4)	114

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

The H atoms in the piperidine ring were refined with a riding model and their  $U_{iso}$  values were set at  $1.2U_{eq}$  of their carrier atoms. The other H atoms were refined isotropically [C—H distances in the range 0.96 (2)–1.00 (2) Å].

Data collection: *Kuma KM-4 Software* (Kuma, 1992). Cell refinement: *Kuma KM-4 Software*. Data reduction: *Kuma KM-4 Software*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *Stereochemical Workstation Operation Manual* (Siemens, 1989). Software used to prepare material for publication: *SHELXL93*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1351). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1999). **C55**, 1952–1955

## Salannin and 3-deacetylsalannin

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(Received 17 March 1999; accepted 29 June 1999)

## Abstract

The crystal structures of two tetranortriterpenoids, salannin [C<sub>34</sub>H<sub>44</sub>O<sub>9</sub>, (I)] and 3-deacetylsalannin [C<sub>32</sub>H<sub>42</sub>O<sub>8</sub>, (II)], are described. The orientation and conformation of the tigloyl (2-methyl-2-butenoyl) group, the carbomethoxy group and ring *E* are different in the two structures. The molecular packing depends on C—H...O hydrogen bonds in (I) and on O—H...O hydrogen bonds in (II). A comparison of the structural features indicates that the conformations of large parts of the molecules are similar to those in azadirachtins.