

*Acta Cryst.* (1995). C51, 2620–2621

### 3-(4-Bromobenzyl)-5-(4-fluorobenzylidene)-imidazolidine-2,4-dione

C. A. DE SIMONE,\* J. ZUKERMAN-SCHPECTOR AND  
M. A. PEREIRA†

*Departamento de Química, Universidade Federal de São Carlos, Caixa Postal 676, 13565 905 São Carlos SP, Brazil*

LUU-DUC CUONG,‡ IVAN R. PITTA, SUELY L. GALDINO AND E. L. CAVALCANTI DE AMORIM

*Laboratório de Planejamento e Síntese de Fármacos, Departamento de Antibióticos, Universidade Federal de Pernambuco, 50739 Recife PE, Brazil*

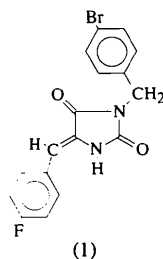
(Received 6 June 1994; accepted 17 May 1995)

#### Abstract

The imidazolidinedione and fluorobenzylidene rings in the title compound, C<sub>17</sub>H<sub>12</sub>BrFN<sub>2</sub>O<sub>2</sub>, are coplanar; the dihedral angle between this plane and the plane of the substituted bromobenzyl ring is 104.9 (2)°. The molecules in the crystal are held together by hydrogen bonds and van der Waals interactions.

#### Comment

The title compound, (1), belongs to a class of imidazolidine and thiazolidine derivatives that display various pharmacological activities, including antibacterial, antifungal and insecticidal activities (Labouta, Salama, Eshba, Kader & El-Chrbini, 1987). As knowledge of its stereochemistry may assist in the understanding of its pharmacological behaviour, a crystal structure determination was undertaken.



The imidazolidinedione and fluorobenzylidene rings are coplanar, with  $\sigma_{av}$  [defined as  $(\sum d_i^2/N-3)^{1/2}$ ] equal to 0.02 Å for the 11 atoms. The molecules are linked *via*

† Permanent address: Departamento de Química, Universidade Federal de Alagoas, 57061 290 Maceió Al, Brazil.

‡ Permanent address: Université Joseph Fourier Grenoble I, Laboratoire de Chimie Pharmacie, France.

an intermolecular hydrogen bond. The N(2)···O(1<sup>i</sup>) and H(N2)···O(1<sup>i</sup>) distances are 2.851 (6) and 1.927 (6) Å, respectively, and the N(2)—H(N2)···O(1<sup>i</sup>) angle is 159.3 (6)° [symmetry code: (i) 1 - x, 1 - y, -1 - z].

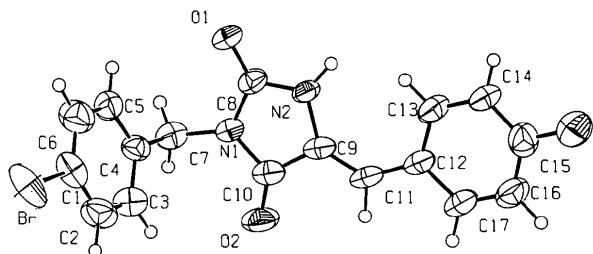


Fig. 1. Projection of C<sub>17</sub>H<sub>12</sub>BrFN<sub>2</sub>O<sub>2</sub> showing the atom-labelling scheme (PLATON; Spek, 1990). Displacement ellipsoids, plotted at the 50% probability level, are shown for the non-H atoms.

#### Experimental

Crystals of 3-(4-bromobenzyl)-5-(4-fluorobenzylidene)imidazolidine-2,4-dione were obtained from cold pentane.

#### Crystal data

C<sub>17</sub>H<sub>12</sub>BrFN<sub>2</sub>O<sub>2</sub>

*M<sub>r</sub>* = 375.20

Triclinic

*P*1

*a* = 5.175 (1) Å

*b* = 11.172 (2) Å

*c* = 14.057 (2) Å

α = 82.46 (1)°

β = 81.70 (2)°

γ = 77.03 (2)°

*V* = 779.5 (3) Å<sup>3</sup>

*Z* = 2

*D<sub>c</sub>* = 1.598 Mg m<sup>-3</sup>

Mo Kα radiation

λ = 0.71073 Å

Cell parameters from 25 reflections

θ = 9–21°

μ = 2.629 mm<sup>-1</sup>

*T* = 292 K

Irregular

0.45 × 0.25 × 0.15 mm

Colourless

#### Data collection

Enraf–Nonius CAD-4 diffractometer

ω/2θ scans

Absorption correction:

refined from Δ*F*

(*DIFABS*; Walker &

Stuart, 1983)

*T<sub>min</sub>* = 0.34, *T<sub>max</sub>* = 0.67

3096 measured reflections

2408 independent reflections

1601 observed reflections

[*I* > 3σ(*I*)]

*R<sub>int</sub>* = 0.015

θ<sub>max</sub> = 25°

*h* = -6 → 6

*k* = -13 → 13

*l* = -1 → 16

2 standard reflections

frequency: 30 min

intensity decay: 0.7%

#### Refinement

Refinement on *F*

*R* = 0.058

*wR* = 0.062

*S* = 1.65

1601 reflections

209 parameters

*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*)  
+ 0.0011|*F<sub>o</sub>*|<sup>2</sup>]

(Δ/σ)<sub>max</sub> = 0.001

Δρ<sub>max</sub> = 0.73 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -0.70 e Å<sup>-3</sup>

Atomic scattering factors from *SHELX76* (Sheldrick, 1976)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )
$$B_{\text{eq}} = (4/3)\sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> <sub>eq</sub>
Br	0.4214 (2)	0.2614 (1)	0.0837 (1)	7.88 (5)
F	0.9791 (9)	0.0089 (3)	-0.8383 (3)	7.2 (2)
O(1)	0.2653 (8)	0.5349 (3)	-0.3994 (3)	4.3 (2)
O(2)	-0.1891 (8)	0.2246 (4)	-0.3658 (3)	5.6 (2)
N(1)	-0.0045 (9)	0.3928 (4)	-0.3623 (3)	3.6 (2)
N(2)	0.3114 (8)	0.3686 (3)	-0.4864 (3)	3.3 (2)
C(1)	0.243 (1)	0.3185 (7)	-0.0273 (4)	5.0 (3)
C(2)	0.095 (1)	0.2450 (6)	-0.0579 (5)	5.7 (4)
C(3)	-0.037 (1)	0.2874 (6)	-0.1370 (5)	5.2 (3)
C(4)	-0.023 (1)	0.4003 (5)	-0.1880 (4)	3.6 (2)
C(5)	0.126 (1)	0.4702 (5)	-0.1558 (5)	4.8 (3)
C(6)	0.260 (1)	0.4296 (7)	-0.0761 (5)	5.8 (4)
C(7)	-0.161 (1)	0.4434 (5)	-0.2763 (5)	4.2 (3)
C(8)	0.200 (1)	0.4414 (5)	-0.4155 (4)	3.5 (2)
C(9)	0.189 (1)	0.2677 (5)	-0.4795 (4)	3.6 (3)
C(10)	-0.026 (1)	0.2867 (5)	-0.3981 (4)	3.9 (3)
C(11)	0.239 (1)	0.1705 (5)	-0.5315 (4)	4.0 (3)
C(12)	0.437 (1)	0.1328 (5)	-0.6104 (4)	3.9 (3)
C(13)	0.634 (1)	0.1969 (5)	-0.6530 (5)	4.6 (3)
C(14)	0.814 (1)	0.1560 (5)	-0.7285 (5)	4.8 (3)
C(15)	0.801 (1)	0.0499 (6)	-0.7633 (5)	5.0 (3)
C(16)	0.612 (1)	-0.0175 (5)	-0.7250 (5)	5.3 (3)
C(17)	0.432 (1)	0.0252 (5)	-0.6497 (5)	4.7 (3)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Br—C(1)	1.893 (6)	F—C(15)	1.352 (8)
O(1)—C(8)	1.224 (6)	O(2)—C(10)	1.212 (7)
N(1)—C(7)	1.459 (8)	N(1)—C(8)	1.377 (7)
N(1)—C(10)	1.379 (7)	N(2)—C(8)	1.350 (7)
N(2)—C(9)	1.398 (7)	C(1)—C(2)	1.38 (1)
C(1)—C(6)	1.35 (1)	C(2)—C(3)	1.36 (1)
C(3)—C(4)	1.378 (9)	C(4)—C(5)	1.369 (8)
C(4)—C(7)	1.492 (8)	C(5)—C(6)	1.38 (1)
C(9)—C(10)	1.477 (8)	C(9)—C(11)	1.345 (8)
C(11)—C(12)	1.435 (8)	C(12)—C(13)	1.396 (9)
C(12)—C(17)	1.394 (8)	C(13)—C(14)	1.361 (9)
C(14)—C(15)	1.359 (9)	C(15)—C(16)	1.37 (1)
C(16)—C(17)	1.37 (1)		
C(7)—N(1)—C(8)	123.7 (4)	N(2)—C(9)—C(10)	105.2 (4)
C(7)—N(1)—C(10)	125.7 (5)	N(2)—C(9)—C(11)	131.4 (5)
C(8)—N(1)—C(10)	110.5 (4)	C(10)—C(9)—C(11)	123.4 (5)
C(8)—N(2)—C(9)	110.5 (4)	O(2)—C(10)—N(1)	125.0 (5)
Br—C(1)—C(2)	119.2 (5)	O(2)—C(10)—C(9)	129.6 (5)
Br—C(1)—C(6)	120.4 (5)	N(1)—C(10)—C(9)	105.4 (5)
C(2)—C(1)—C(6)	120.4 (6)	C(9)—C(11)—C(12)	132.7 (5)
C(1)—C(2)—C(3)	119.0 (6)	C(11)—C(12)—C(13)	125.1 (5)
C(2)—C(3)—C(4)	121.8 (6)	C(11)—C(12)—C(17)	118.2 (5)
C(3)—C(4)—C(5)	117.6 (5)	C(13)—C(12)—C(17)	116.6 (5)
C(3)—C(4)—C(7)	121.1 (5)	C(12)—C(13)—C(14)	121.8 (6)
C(5)—C(4)—C(7)	121.3 (5)	C(13)—C(14)—C(15)	119.0 (6)
C(4)—C(5)—C(6)	121.7 (6)	F—C(15)—C(14)	119.2 (6)
C(1)—C(6)—C(5)	119.6 (7)	F—C(15)—C(16)	118.5 (6)
N(1)—C(7)—C(4)	112.2 (5)	C(14)—C(15)—C(16)	122.2 (6)
O(1)—C(8)—N(1)	124.6 (5)	C(15)—C(16)—C(17)	118.0 (6)
O(1)—C(8)—N(2)	127.1 (5)	C(12)—C(17)—C(16)	122.3 (6)
N(1)—C(8)—N(2)	108.3 (4)		

Data were corrected for Lp effects. The structure was solved by direct methods. H atoms were found in difference syntheses and included as fixed contributors with an overall isotropic displacement parameter that refined to  $U_{\text{iso}} = 0.067 (5) \text{\AA}^2$ . The refinement was by blocked-matrix least-squares methods. Programs used were: *SHELXS86* (Sheldrick, 1985) and *SHELX76* (Sheldrick, 1976). Most of the calculations were performed on a VAX 6420 computer at the Instituto de Física e Química de São Carlos.

This work has received partial support from FAPESP (Proc. 94/1213-5), CNPq, FAPEAL and FINEP.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PT1003). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Labouta, I. M., Salama, H. M., Eshba, N. H., Kader, O. & El-Chrbini, E. (1987). *Eur. J. Med. Chem.* **22**, 485–489.  
 Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.  
 Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.  
 Spek, A. L. (1990). *Acta Cryst.* **A46**, C-34.  
 Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.

*Acta Cryst.* (1995). **C51**, 2621–2624

## 8,16-Methano-16H-dinaphtho[2,1-d:1',2'-g]-[1,3]dioxocine-2,14-diol

HERBERT E. KLEI

*Department of Molecular and Cell Biology (U-125), University of Connecticut, Storrs, Connecticut 06269-3125, USA*

ERNESTO CALLEGARI AND J. MICHAEL EDWARDS

*Section of Medicinal Chemistry and Pharmacognosy (U-92), School of Pharmacy, University of Connecticut, Storrs, Connecticut 06269-2092, USA*

JUDITH A. KELLY

*Department of Molecular and Cell Biology (U-125), University of Connecticut, Storrs, Connecticut 06269-3125, USA*

(Received 14 April 1994; accepted 8 March 1995)

## Abstract

The symmetry of the chemical structure of the title compound,  $\text{C}_{23}\text{H}_{16}\text{O}_4$ , is not preserved in the crystal structure; two conformational isomers are present as required by the centrosymmetry of the space group. The naphthalene components are splayed away from each other and twisted in order to separate the aromatic H atoms H14 and H26. The chevron-shaped molecules are stacked on top of one another. Four such stacks (two antiparallel pairs) are apparent when the unit cell is viewed along the *z* axis. Both the conformation of