Table 2. *Selected geometric parameters (Å)*

C1—C2	1.345 (3)	C8—C9	1.374 (3)
C1—O13	1.350 (3)	C9—O13	1.372 (2)
C2—C3	1.464 (3)	C1′—C2′	1.405 (3)
C2—C1′	1.486 (3)	C1′—C6′	1.387 (3)
C3—C4	1.446 (3)	C2′—C3′	1.380 (3)
C3—O17	1.247 (3)	C3′—C4′	1.405 (3)
C4—C5	1.415 (3)	C3′—O19	1.370 (3)
C4—C9	1.401 (3)	C4′—C5′	1.373 (3)
C5—C6	1.381 (3)	C4′—O18	1.366 (2)
C5—O16	1.349 (3)	C5′—C6′	1.390 (3)
C6—C7	1.404 (3)	C10—O15	1.422 (4)
C6—O15	1.377 (2)	C11—O19	1.429 (3)
C7—C8	1.384 (3)	C12—O18	1.417 (3)
C7—O14	1.347 (2)		

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2′—H2′···O17	0.94 (2)	2.32 (2)	2.880 (2)	118 (2)
C10—H10A···O16	0.95 (4)	2.64 (4)	3.159 (3)	115 (3)
O14—H14···O15	0.86 (4)	2.28 (4)	2.725 (2)	113 (3)
O16—H16···O17	1.00 (3)	1.64 (3)	2.562 (2)	150 (3)
C8—H8···O14^{i}	0.88 (3)	2.68 (3)	3.346 (3)	133 (2)
C5′—H5′···O16^{ii}	0.94 (2)	2.73 (2)	3.655 (3)	170 (2)
C12—H12B···O15^{ii}	0.97 (2)	2.77 (3)	3.373 (3)	121 (2)
C11—H11B···O15^{iii}	1.03 (3)	2.75 (3)	3.636 (3)	145 (2)
O16—H16···O16^{iii}	1.00 (3)	2.64 (3)	3.135 (2)	110 (2)
C11—H11C···O14^{iv}	1.00 (3)	2.72 (3)	3.621 (4)	150 (2)
O14—H14···O18^{v}	0.86 (4)	2.46 (4)	3.022 (2)	123 (3)
O14—H14···O19^{v}	0.86 (4)	2.13 (3)	2.871 (2)	145 (2)

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

The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985). The initial *R* factor for the model proposed was 0.19. After a few cycles of full-matrix least-squares refinement, the *R* factor reduced to 0.11. All H atoms were located from the difference Fourier map and were refined isotropically.

Data collection: *Enraf–Nonius CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *SDP* (Frenz, 1978). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: DE1027). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acetoxymethyl 4-Chloro-*N*-furfuryl-5-sulfamoylanthranilate, an Absorption Furosemide Prodrug

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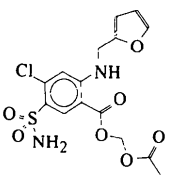
Abstract

The title compound, $C_{15}H_{15}ClN_2O_7S$, which was synthesized and characterized as the acetoxymethyl ester of 4-chloro-*N*-furfuryl-5-sulfamoylanthranilic acid (furosemide) is an absorption furosemide prodrug. The molecule crystallized in a triclinic unit cell, space group $P\bar{1}$. The crystal structure is stabilized by one intramolecular and two intermolecular hydrogen bonds.

Comment

Furosemide is a strong diuretic agent widely used in hypertensive crisis. The use of some acyloxymethyl esters of furosemide as prodrugs to improve the therapeutic success of this drug has been studied by Prandi, Fagiolino, Manta & Llera (1992).



Prol

The structure described in this paper is the acetoxymethyl prodrug, Pro1, which has been synthesized and spectroscopically characterized (Prandi, Fagiolino, Manta, Llera, Aiache & Couquelet, 1992). This work is part of a project involving research on the structures of furosemide prodrugs.

The Pro1 molecule contains a six-membered aromatic ring (atoms C1–C6) with which atoms N1, H1, C7 and O1 are coplanar (C7 and O1 are part of the carboxylic group esterified with the acetoxymethyl group). The equation of the plane is $3.739(40)x + 7.300(8)y + 9.979(14)z = 3.756(21)$ and the maximum deviation is −0.082 (10) Å for O1. There is an intramolecular hydrogen bond between N1 and O1 as shown by the N1···O1 [2.713 (5) Å] and H1···O1 [1.95 (5) Å] distances. This hydrogen bond and the high π–π overlap possibilities between the aromatic ring and the carboxylic group may explain the planarity of this part of the molecule. The interatomic distances and conformation are very similar to those of furosemide (Lamotte, Campsteyn, Dupont & Vermeire, 1978) except for the torsion angles linking the six-membered aromatic and furan rings. Relevant torsion angles are listed in Table 3; these demonstrate that furosemide and its prodrug have considerable conformational flexibility about the N1—C11 and C11—C12 bonds. The bond distances between the benzene ring C atoms in Pro1 suggest a decrease in its aromatic character. As in the crystal structure of furosemide, this decrease may be attributed to the presence of the sulfamoyl group.

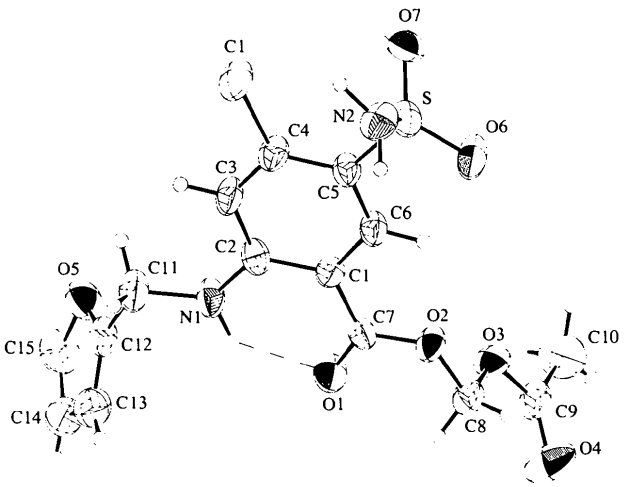


Fig. 1. *ZORTEP* (Zsolnai & Pritzkow, 1995) drawing of Pro1. The intramolecular hydrogen bond is marked as a dashed line. Displacement ellipsoids are drawn at the 50% probability level and H atoms as spheres of arbitrary radii.

The presence of the acetoxymethyl group has the main effect on the packing of Pro1 compared to that of furosemide. This could be explained by the absence of the acidic H atom of furosemide and by the presence of the acetate carbonyl-O atom, O4. There are intermolecular hydrogen bonds between both H atoms at N2 and both ester carbonyl-O atoms, O1 and O4. Therefore, O1 is involved in one intermolecular and one intramolecular hydrogen bond. Pro1 packs as a dimeric unit about an inversion centre with the dimers stabilized by two symmetry-equivalent hydrogen bonds between N2 of one molecule and O1 of the other. These dimers are linked in infinite chains by intermolecular hydrogen bonds between N2 and O4. The molecular geometry of these hydrogen bonds is described in Table 4 and the packing is illustrated in Fig. 2.

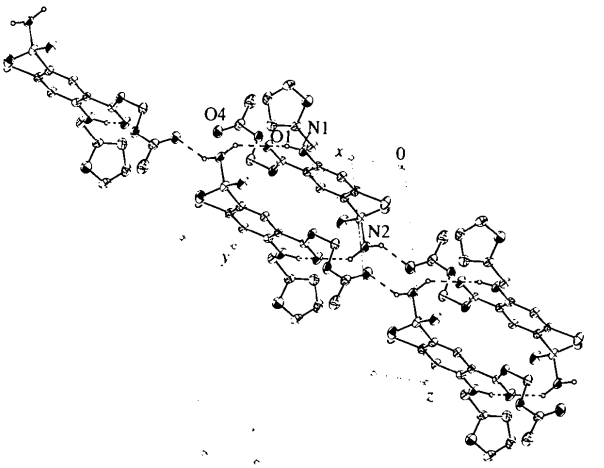


Fig. 2. *ZORTEP* drawing of the chain of Pro1 molecules showing the hydrogen-bonding scheme and unit cell. Most H atoms are omitted for clarity. Ellipsoids are drawn at the 30% probability level.

Experimental

Pro1 was obtained as previously described (Prandi, Fagiolino, Manta, Llera, Aiache, & Couquelet, 1992) and crystallization was performed by vapour diffusion (ethyl acetate/hexane) at room temperature.

Crystal data

$C_{15}H_{15}ClN_2O_7S$
$M_r = 402.80$
Triclinic
$P\bar{1}$
$a = 8.502(2)$ Å
$b = 9.653(3)$ Å
$c = 11.767(2)$ Å
$\alpha = 72.01°$
$\beta = 74.57°$
$\gamma = 72.74°$
$V = 860.8(4)$ Å^3
$Z = 2$
$D_x = 1.554$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 40 reflections
$\theta = 7.5$–$15°$
$\mu = 0.385$ mm^{-1}
$T = 293(2)$ K
Prismatic
0.17 × 0.14 × 0.08 mm
Colourless

Data collection

Siemens *R3m* diffractometer	$\theta_{max} = 24.05°$
$\theta/2\theta$ scans	$h = -3 \rightarrow 9$
Absorption correction: none	$k = -11 \rightarrow 11$
4213 measured reflections	$l = -13 \rightarrow 13$
2722 independent reflections	2 standard reflections
1461 observed reflections $[I > 2\sigma(I)]$	monitored every 98 reflections
$R_{int} = 0.0243$	intensity decay: none

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.0511$	$\Delta\rho_{max} = 0.298$ e Å^{-3}
$wR(F^2) = 0.1179$	$\Delta\rho_{min} = -0.449$ e Å^{-3}
$S = 1.088$	Extinction correction: none
2722 reflections	Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
269 parameters	
H atoms refined isotropically	
$w = 1/[\sigma^2(F_o^2) + (0.0687P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

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

	x	y	z	U_{eq}
C1	0.5006 (6)	0.4033 (5)	−0.1054 (4)	0.0335 (12)
C2	0.6547 (6)	0.2968 (5)	−0.0865 (4)	0.0380 (12)
C3	0.6462 (7)	0.1662 (6)	0.0104 (4)	0.0419 (14)
C4	0.4962 (6)	0.1429 (5)	0.0816 (4)	0.0398 (13)
C5	0.3443 (6)	0.2507 (5)	0.0662 (4)	0.0371 (12)
C6	0.3500 (6)	0.3784 (5)	−0.0275 (4)	0.0376 (13)
C7	0.4984 (6)	0.5405 (5)	−0.2055 (4)	0.0344 (12)
O1	0.6199 (4)	0.5714 (4)	−0.2821 (3)	0.0530 (10)
O2	0.3457 (4)	0.6354 (3)	−0.2026 (3)	0.0434 (9)
C8	0.3254 (7)	0.7637 (5)	−0.3003 (4)	0.0459 (15)
O3	0.2909 (5)	0.7158 (4)	−0.3927 (3)	0.0496 (10)
C9	0.2515 (7)	0.8236 (6)	−0.4914 (5)	0.0500 (15)
O4	0.2549 (6)	0.9510 (4)	−0.5056 (3)	0.0751 (14)
C10	0.2022 (11)	0.7653 (9)	−0.5758 (7)	0.081 (2)
N1	0.8048 (5)	0.3166 (5)	−0.1581 (4)	0.0467 (12)
C11	0.9666 (6)	0.2225 (6)	−0.1361 (5)	0.0509 (15)
C12	1.1006 (6)	0.2629 (6)	−0.2421 (5)	0.0431 (13)
C13	1.1766 (8)	0.3764 (7)	−0.2864 (6)	0.058 (2)
C14	1.2891 (8)	0.3522 (7)	−0.3950 (6)	0.061 (2)
C15	1.2745 (8)	0.2281 (8)	−0.4101 (5)	0.061 (2)
O5	1.1573 (5)	0.1685 (4)	−0.3185 (4)	0.0574 (10)
S	0.1468 (2)	0.2306 (2)	0.15933 (12)	0.0447 (4)
O6	0.0319 (4)	0.3679 (4)	0.1196 (3)	0.0604 (11)
O7	0.1150 (5)	0.0940 (4)	0.1583 (3)	0.0594 (11)
N2	0.1540 (6)	0.2188 (5)	0.2983 (4)	0.0509 (12)
Cl	0.5000 (2)	−0.02080 (15)	0.19650 (13)	0.0593 (5)

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

C1—C6	1.400 (6)	C1—C2	1.427 (6)
C1—C7	1.475 (6)	C2—N1	1.359 (6)
C2—C3	1.420 (6)	C3—C4	1.366 (7)
C4—C5	1.412 (6)	C4—Cl	1.732 (5)
C5—C6	1.380 (6)	C5—S	1.772 (5)
C7—O1	1.220 (5)	C7—O2	1.347 (5)
O2—C8	1.409 (5)	C8—O3	1.429 (6)
O3—C9	1.340 (6)	C9—O4	1.197 (6)
C9—C10	1.479 (9)	N1—C11	1.445 (6)
C11—C12	1.487 (7)	C12—C13	1.333 (7)
C12—O5	1.373 (6)	C13—C14	1.416 (8)
C14—C15	1.309 (9)	C15—O5	1.367 (6)
S—O6	1.422 (3)	S—O7	1.427 (4)
S—N2	1.620 (5)		
C6—C1—C7	119.5 (4)	C2—C1—C7	120.7 (4)
N1—C2—C3	120.4 (4)	N1—C2—C1	122.3 (4)
C3—C4—Cl	117.4 (4)	C5—C4—Cl	121.0 (4)
C6—C5—S	118.2 (4)	C4—C5—S	123.8 (3)
O1—C7—O2	121.7 (4)	O1—C7—C1	125.5 (4)
O2—C7—C1	112.8 (4)	C7—O2—C8	117.6 (4)
O2—C8—O3	105.3 (4)	C9—O3—C8	116.0 (4)
O4—C9—O3	122.5 (6)	O4—C9—C10	125.7 (5)
O3—C9—C10	111.8 (5)	C2—N1—C11	125.1 (4)
N1—C11—C12	110.3 (4)	C13—C12—O5	109.6 (5)
C13—C12—C11	135.0 (6)	O5—C12—C11	115.3 (5)
C12—C13—C14	106.9 (6)	C15—C14—C13	106.9 (6)
C14—C15—O5	111.0 (6)	C15—O5—C12	105.6 (5)
O6—S—O7	118.8 (2)	O6—S—N2	106.8 (3)
O7—S—N2	107.2 (2)		
C7—C1—C2—N1	0.5 (7)	Cl—C4—C5—S	−0.3 (7)
C2—C3—C4—Cl	179.5 (4)	C7—C1—C6—C5	178.7 (5)
S—C5—C6—C1	−180.0 (4)	C6—C1—C7—O2	5.7 (6)
C2—C1—C7—O1	4.0 (8)	C7—O2—C8—O3	84.5 (5)
O1—C7—O2—C8	8.0 (7)	C8—O3—C9—O4	4.5 (8)
O2—C8—O3—C9	174.1 (4)	C3—C2—N1—C11	−7.9 (8)
C2—N1—C11—C12	171.4 (5)	N1—C11—C12—C13	73.9 (8)
C12—C13—C14—C15	0.3 (7)	O5—C12—C13—C14	−1.1 (6)
C13—C12—O5—C15	1.4 (6)	C14—C15—O5—C12	−1.2 (7)
C6—C5—S—O7	123.7 (4)	C6—C5—S—O6	−5.3 (5)
N1—C2—C3—C4	−178.6 (5)	C6—C5—S—N2	−119.4 (4)

Table 3. *Torsion angles in furosemide and Pro1 (°)*

Torsion Angles	Pro1	Furosemide* Molecule *A*	Furosemide* Molecule *B*
C3—C2—N1—C11	−7.9 (8)	−15.2	5.3
C1—C2—N1—C11	172.7 (5)	165.5	−175.1
C2—N1—C11—C12	171.4 (5)	−62.9	83.5
N1—C11—C12—C13	73.9 (8)	127.6	116.7
N1—C11—C12—O5	−101.9 (5)	−54.0	−67.1

* Standard deviations for torsion angles of furosemide were not available.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.97 (5)	2.713 (5)	1.95 (5)	135 (4)
N2—H2···O1^{i}	1.03 (5)	3.116 (6)	2.31 (5)	135 (4)
N2—H3···O4^{ii}	1.00 (5)	2.996 (6)	2.10 (5)	144 (4)

Symmetry codes: (i) $1 - x, 1 - y, -z$; (ii) $x, y - 1, 1 + z$.

The structure was solved by the Patterson method which located S and Cl atoms. The rest of the non-H atoms were located by difference Fourier maps and refined anisotropically. All H atoms were located by difference Fourier maps and refined isotropically with an isotropic displacement parameter (U_{iso}) equal to $1.2U_{eq}$ of the parent atom. Both H atoms at C8 were refined with fixed orientations and both H atoms at C11 were refined as riding atoms in order to improve distances.

Data collection: *P3/P4/PC* (Siemens, 1991). Cell refinement: *P3/P4/PC*. Data reduction: *XDISK* in *SHELXTL/PC* (Sheldrick, 1990*a*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*b*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai & Pritzkow, 1995). Software used to prepare material for publication: *CIFTAB* in *SHELXL*93.

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Universidad de la República, Uruguay) and CONICYT (Consejo Nacional de Investigación Científica y Tecnológica, Uruguay) for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SX1003). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-(*p*-Diethylaminobenzylidene)-1,3-indandione

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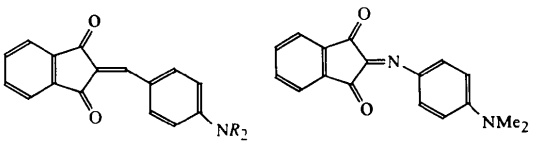
Abstract

The title compound, $C_{20}H_{19}NO_2$, belongs to the class of donor–acceptor-substituted conjugated polyenes. Within the structure, O atoms are situated in the plane of the 1,3-indandione fragment and the N atom lies in the plane of the *p*-benzylidene fragment which forms a dihedral angle of 7.6 (2)° with the indandione nucleus.

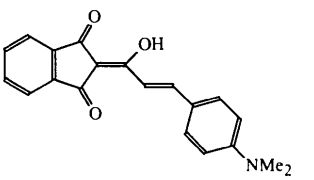
Comment

2-(*p*-Dimethylaminobenzylidene)-1,3-indandione, (I*b*), exhibits a series of unusual solid-state and solution photophysical properties, in particular non-linear optical behaviour and recently discovered non-linear fluorescent properties (Valkunas *et al.*, 1993, and references therein). When this derivative absorbs at 450–500 nm in solution, it forms three deeply coloured polymorphs: the most-stable dark-red α-modification (space group $P2_1/c$, Z = 8; Magomedova & Zvonkova, 1978), a less-stable blue-coloured β-modification (space group $P2_1/c$, Z = 4; Magomedova, Zvonkova, Neigaus & Novakovskaya, 1980), and the least-stable red prisms of the γ-modification (non-centrosymmetric space group $Pna2_1$, Z = 4; Magomedova & Zvonkova, 1980).



(I*a*) R = Et
(I*b*) R = Me

(II)



(III)

During our investigations on the synthesis and properties of structural analogues of (I*b*), we have found that the *p*-diethylamino derivative, the title compound (I*a*), always crystallizes as deep-green lustrous crystals, whereas its UV–visible absorption properties in solution (λ_{max} = 488 nm in dichloromethane) are practically the same as those of (I*b*). Solid-state investigations of this derivative may shed light on unusual polychromic properties of the whole series of electron-donor-substituted derivatives of 2-ylidene-1,3-indandiones.

The 1,3-indandione moiety of (I*a*) is planar [r.m.s. Δ = 0.012 (4) Å] and the two O atoms are displaced by 0.028 (5) (O1) and 0.059 (5) Å (O2) from the plane. The N atom is displaced from the plane of the *p*-phenyl ring [r.m.s. Δ = 0.003 (3) Å] by 0.12 (8) Å. The bond lengths within the bridge linking the 1,3-indandione accepting and diethylamino donating moieties exhibit the presence of considerable conjugation. Thus, the aromatic C12—C13 [1.376 (6) Å] and C15—C16 [1.373 (6) Å] bonds are slightly shorter than other aromatic bonds in the *p*-phenylene ring [1.396 (6)–1.400 (6) Å], indicating the *p*-quinoid character. All of the above-mentioned geometrical features are also typical of (I*b*), (II) [2-(4′-dimethylaminophenylimino)-1,3-indandione; Magomedova, Zvonkova, Geita, Novakovskaya, Neigaus & Belsky, 1980] and (III) [2-(4′-dimethylaminocinnamoyl)-1,3-indandione; Magomedova, Zvonkova, Geita, Smelyanskaya & Ginzburg, 1980].