

## Refinement

Refinement on *F**R* = 0.046*wR* = 0.072*S* = 1.43

1840 reflections

154 parameters

H atoms were included

in the structure-factor

calculations at fixed

positions with C—H =

0.95 Å

$$w = 1/[\sigma^2(F) + (0.04F)^2]$$

$$(\Delta/\sigma)_{\max} = 0.03$$

$$\Delta\rho_{\max} = 0.22 \text{ e } \text{Å}^{-3}$$

$$\Delta\rho_{\min} = -0.15 \text{ e } \text{Å}^{-3}$$

Atomic scattering factors

from *International Tables*for *X-ray Crystallography*

(1974, Vol. IV, Table

2.2B)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>eq</sub>
O(1)	0.4615 (2)	0.8721 (1)	0.1527 (2)	0.0978 (5)
O(2)	0.9159 (1)	0.85509 (6)	0.8481 (1)	0.0504 (3)
O(3)	1.0664 (1)	0.93121 (6)	0.7265 (1)	0.0598 (4)
C(1)	0.8336 (2)	0.86594 (8)	0.5612 (2)	0.0404 (4)
C(2)	0.8426 (2)	0.91508 (9)	0.4218 (2)	0.0531 (5)
C(3)	0.7603 (3)	0.8857 (1)	0.2588 (2)	0.0655 (5)
C(4)	0.5842 (3)	0.8565 (1)	0.2549 (2)	0.0613 (5)
C(5)	0.5703 (2)	0.80745 (9)	0.3882 (2)	0.0550 (5)
C(6)	0.6438 (2)	0.84245 (8)	0.5514 (2)	0.0422 (4)
C(7)	0.5260 (2)	0.9009 (1)	0.5847 (2)	0.0552 (5)
C(8)	0.5950 (2)	0.9355 (1)	0.7431 (2)	0.0632 (5)
C(9)	0.7778 (2)	0.96122 (9)	0.7524 (2)	0.0588 (5)
C(10)	0.8976 (2)	0.90382 (8)	0.7212 (2)	0.0432 (4)
C(11)	0.9534 (2)	0.8043 (1)	0.5562 (2)	0.0617 (5)
C(12)	0.3883 (3)	0.7788 (1)	0.3735 (3)	0.0865 (6)
C(13)	1.0684 (2)	0.8728 (1)	0.9620 (2)	0.0599 (5)
C(14)	1.1795 (2)	0.9087 (1)	0.8675 (2)	0.0585 (5)

Table 2. Selected geometric parameters (Å, °)

O(1)—C(4)	1.205 (2)	C(3)—C(4)	1.496 (3)
O(2)—C(10)	1.428 (2)	C(4)—C(5)	1.510 (3)
O(2)—C(13)	1.428 (2)	C(5)—C(6)	1.556 (2)
O(3)—C(10)	1.427 (2)	C(5)—C(12)	1.522 (3)
O(3)—C(14)	1.417 (2)	C(6)—C(7)	1.533 (2)
C(1)—C(2)	1.543 (2)	C(7)—C(8)	1.513 (2)
C(1)—C(6)	1.552 (2)	C(8)—C(9)	1.514 (3)
C(1)—C(10)	1.548 (2)	C(9)—C(10)	1.523 (2)
C(1)—C(11)	1.534 (2)	C(13)—C(14)	1.483 (3)
C(2)—C(3)	1.526 (2)		
C(10)—O(2)—C(13)	107.4 (1)	C(6)—C(5)—C(12)	114.1 (2)
C(10)—O(3)—C(14)	109.5 (1)	C(1)—C(6)—C(5)	111.0 (1)
C(2)—C(1)—C(6)	109.6 (1)	C(1)—C(6)—C(7)	112.6 (1)
C(2)—C(1)—C(10)	109.3 (1)	C(5)—C(6)—C(7)	111.3 (1)
C(2)—C(1)—C(11)	109.8 (1)	C(6)—C(7)—C(8)	112.1 (1)
C(6)—C(1)—C(10)	109.1 (1)	C(7)—C(8)—C(9)	111.2 (2)
C(6)—C(1)—C(11)	111.1 (1)	C(8)—C(9)—C(10)	111.4 (1)
C(10)—C(1)—C(11)	107.8 (1)	O(2)—C(10)—O(3)	106.0 (1)
C(1)—C(2)—C(3)	113.4 (1)	O(2)—C(10)—C(1)	108.7 (1)
C(2)—C(3)—C(4)	112.6 (2)	O(2)—C(10)—C(9)	109.0 (1)
O(1)—C(4)—C(3)	121.8 (2)	O(3)—C(10)—C(1)	109.9 (1)
O(1)—C(4)—C(5)	122.5 (2)	O(3)—C(10)—C(9)	109.2 (1)
C(3)—C(4)—C(5)	115.7 (1)	C(1)—C(10)—C(9)	113.8 (1)
C(4)—C(5)—C(6)	109.2 (1)	O(2)—C(13)—C(14)	104.6 (1)
C(4)—C(5)—C(12)	112.4 (1)	O(3)—C(14)—C(13)	105.7 (1)

The structure was determined by direct methods using *MolEN* (Fair, 1990). Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: local program. Data reduction: *MolEN*. Program(s) used to refine structure: *MolEN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances and angles involving non-H atoms and torsion angles have been deposited with the IUCr (Reference: BK1068). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
- Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
- Heathcock, C. H., Ratcliffe, R. & Van, J. (1972). *J. Org. Chem.* **37**, 1796–1807.
- Huffman, J. W. & Balke, W. H. (1988). *J. Org. Chem.* **53**, 3828–3831.
- Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- Park, K., Scott, W. J. & Wiemer, D. F. (1994). *J. Org. Chem.* In the press.
- Schmitz, F. J., Lakshmi, V., Powell, D. R. & van der Helm, D. J. (1984). *J. Org. Chem.* **49**, 241–244.

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## Oxime of 1-Methyl-3-trifluoroacetyl-pyrrolidin-2-one

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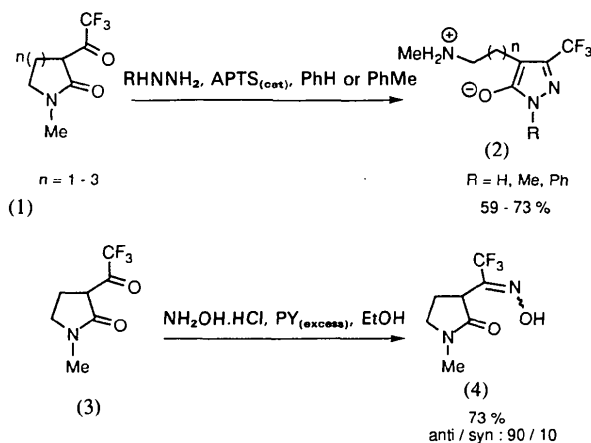
## Abstract

The title compound, 3-(1-hydroxyimino-2,2,2-trifluoroethyl)-1-methylpyrrolidin-2-one, C<sub>7</sub>H<sub>9</sub>F<sub>3</sub>N<sub>2</sub>O<sub>2</sub>, is obtained by condensation of 1-methyl-3-trifluoroacetylpyrrolidin-2-one and hydroxylamine without ring opening of the lactam. The least-squares planes through the pyrrolidine and oxime groups are approximately perpendicular to each other.

### Comment

As part of our ongoing search for new methods of preparation of trifluoromethylated heterocycles (Bouillon, Atès, Janousek & Viehe, 1993), we have examined the question of whether condensations of 3-trifluoroacetylactams (Bouillon, Frisque-Hesbain, Janousek & Viehe, 1994) or 3-trifluoroacetylbenzolactams (Bouillon, Janousek, Viehe, Tinant & Declercq, 1994) with 1,2-bis-nucleophiles would proceed with or without opening of the lactam structure.

The 3-trifluoroacetylactams (1) reacted with hydrazines to produce the zwitterionic forms of the 5-hydroxy-3-trifluoromethylpyrazoles (2). We were intrigued by the question of whether hydroxylamine would give such opening of the lactam structure. In contrast to hydrazines, the reaction of 1-methyl-3-trifluoroacetylpyrrolidin-2-one, (3), furnished the title compound, (4), after chromatography on silica gel; both *anti* and *syn* stereoisomers were obtained in the ratio 90/10. As shown in Fig. 1, the isomer which crystallized from a petroleum ether dichloromethane mixture has the *anti* configuration.



The pyrrolidin-2-one (4) has a flat envelope conformation ( $\Delta C_s = 2.7^\circ$ ) with the mirror plane of symmetry through  $C_4$ . The dihedral angle between the oxime

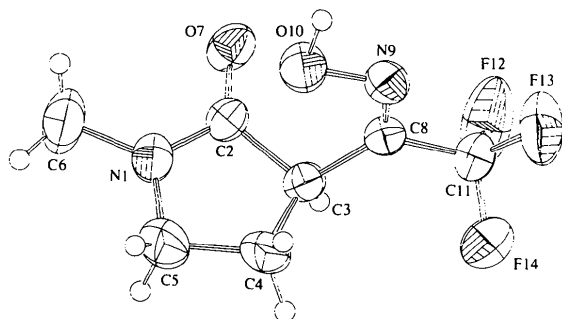


Fig. 1. PLATON (Spek, 1990) drawing of the molecule (4) with atomic numbering. The displacement ellipsoids are drawn at the 50% probability level.

and ring least-squares planes is  $80(1)^\circ$ . The  $C=N$  and  $N-O$  bond lengths of 1.259(4) and 1.386(3) Å, respectively, are shorter than the expected values: 1.281(13) and 1.416(6) Å (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). There is an intermolecular hydrogen bond between the lactam  $C=O$  and the oxime  $O-H$  groups [ $O10-H \cdots O7^i$ :  $O \cdots O$  2.616(4),  $H \cdots O$  1.54(6) Å,  $O-H \cdots O$   $171(2)^\circ$ ; symmetry code: (i)  $\frac{1}{2} + x, \frac{1}{2} - y, z$ ].

### Experimental

The full details of the synthesis are given by Bouillon (1994). Crystals were obtained by slow evaporation at room temperature of a saturated solution of (4) in a mixture of dichloromethane and petroleum ether (1:1).

#### Crystal data

$C_7H_9F_3N_2O_2$   
 $M_r = 210.16$   
 Monoclinic  
 $P2_1/a$   
 $a = 9.685(3)$  Å  
 $b = 7.128(2)$  Å  
 $c = 13.572(4)$  Å  
 $\beta = 99.78(2)^\circ$   
 $V = 923.3(5)$  Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.512$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
 $\lambda = 0.71069$  Å  
 Cell parameters from 21 reflections  
 $\theta = 5-15^\circ$   
 $\mu = 0.149$  mm<sup>-1</sup>  
 $T = 293(2)$  K  
 Parallelepiped  
 $0.36 \times 0.32 \times 0.18$  mm  
 Colourless

#### Data collection

Huber four-circle diffractometer  
 $\omega$  scans  
 Absorption correction: none  
 2119 measured reflections  
 2119 independent reflections  
 1304 observed reflections  
 $[I > 2\sigma(I)]$

$\theta_{max} = 27.50^\circ$   
 $h = 0 \rightarrow 12$   
 $k = 0 \rightarrow 9$   
 $l = -17 \rightarrow 17$   
 1 standard reflection monitored every 50 reflections  
 intensity decay: none

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.073$   
 $wR(F^2) = 0.230$   
 $S = 1.162$   
 2118 reflections  
 134 parameters  
 $w = 1/[\sigma^2(F_o^2) + (0.1751P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.557$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.328$  e Å<sup>-3</sup>  
 Extinction correction: none  
 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<sup>2</sup>)

	$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$			
	x	y	z	$U_{eq}$
N1	0.1506(3)	-0.1790(4)	0.3546(2)	0.0561(7)
C2	0.1133(3)	-0.0089(4)	0.3203(2)	0.0425(7)
C3	0.1254(3)	-0.0006(4)	0.2097(2)	0.0461(7)

C4	0.2059 (5)	-0.1765 (5)	0.1925 (3)	0.0699 (11)
C5	0.1961 (5)	-0.3039 (5)	0.2821 (3)	0.0780 (12)
C6	0.1376 (6)	-0.2443 (6)	0.4548 (3)	0.0871 (13)
O7	0.0708 (2)	0.1153 (3)	0.3695 (2)	0.0555 (6)
C8	0.1922 (3)	0.1776 (4)	0.1806 (2)	0.0441 (7)
N9	0.3044 (3)	0.2517 (3)	0.2236 (2)	0.0492 (7)
O10	0.3667 (2)	0.1507 (3)	0.3065 (2)	0.0572 (6)
C11	0.1243 (4)	0.2791 (5)	0.0882 (2)	0.0527 (8)
F12	0.0112 (3)	0.3654 (5)	0.0996 (2)	0.1152 (12)
F13	0.2047 (3)	0.4091 (4)	0.0577 (2)	0.0936 (9)
F14	0.0942 (4)	0.1644 (4)	0.0121 (2)	0.1173 (12)

Table 2. Selected geometric parameters (Å, °)

N1—C2	1.327 (4)	C4—C5	1.534 (5)
N1—C5	1.450 (4)	C8—N9	1.259 (4)
N1—C6	1.462 (4)	C8—C11	1.500 (4)
C2—O7	1.222 (4)	N9—O10	1.386 (3)
C2—C3	1.527 (4)	C11—F12	1.288 (4)
C3—C8	1.508 (4)	C11—F14	1.311 (4)
C3—C4	1.515 (5)	C11—F13	1.321 (4)
C2—N1—C5	114.7 (3)	N9—C8—C11	114.0 (3)
C2—N1—C6	123.8 (3)	N9—C8—C3	127.4 (3)
C5—N1—C6	121.3 (3)	C11—C8—C3	118.6 (3)
O7—C2—N1	124.6 (3)	C8—N9—O10	112.4 (2)
O7—C2—C3	126.8 (3)	F12—C11—F14	108.2 (4)
N1—C2—C3	108.6 (3)	F12—C11—F13	105.2 (3)
C8—C3—C4	113.7 (3)	F14—C11—F13	104.7 (3)
C8—C3—C2	113.4 (2)	F12—C11—C8	113.0 (3)
C4—C3—C2	104.3 (3)	F14—C11—C8	111.4 (3)
C3—C4—C5	105.3 (3)	F13—C11—C8	113.7 (3)
N1—C5—C4	104.0 (3)		
C5—N1—C2—C3	2.2 (4)	C2—N1—C5—C4	8.8 (5)
N1—C2—C3—C4	-12.4 (3)	C3—C4—C5—N1	-15.9 (4)
C2—C3—C4—C5	17.0 (4)		

The relatively high *R* values are probably the result of poor crystal quality. The shapes of the C6, O7 and, especially, F12 and F13 displacement ellipsoids (Fig. 1) indicate high thermal agitation and unresolved disorder, which may contribute to the high *R* values. The positions of H3 and H10 were determined from a  $\Delta\rho$  map; their site coordinates were refined. All other H atoms were located in calculated positions and refined riding on the attached C atom. A common isotropic displacement parameter for all H atoms was refined [ $U = 0.121 (7) \text{ \AA}^2$ ].

Data collection: local program. Cell refinement: local program. Data reduction: local program. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *PLATON* (Spek, 1990). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1223). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.  
 Bouillon, J.-P. (1994). PhD thesis, Univ. Catholique de Louvain, Belgium.  
 Bouillon, J.-P., Atès, C., Janousek, Z. & Viehe, H. G. (1993). *Tetrahedron Lett.* **34**, 5075–5078.

- Bouillon, J.-P., Frisque-Hesbain, A. M., Janousek, Z. & Viehe, H. G. (1994). *Heterocycles*. In the press.  
 Bouillon, J.-P., Janousek, Z., Viehe, H. G., Tinant, B. & Declercq, J.-P. (1994). *Bull. Soc. Chim. Belg.* In the press.  
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.  
 Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.  
 Spek, A. L. (1990). *Acta Cryst.* **A46**, C-34.

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## Aversin

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## Abstract

The X-ray crystal structure analysis of the mould metabolite (3*aS-cis*)-2,3,3*a*,12*a*-tetrahydro-4-hydroxy-6,8-dimethoxyanthra[2,3-*b*]furo[3,2-*d*]furan-5,10-dione, which contains two C<sub>20</sub>H<sub>16</sub>O<sub>7</sub> molecules per asymmetric unit in *P2*<sub>1</sub>, reveals that the molecule contains five fused rings: a planar section of three six-membered rings and one five-membered ring, with a final *cis*-fused five-membered ring. The OH group in each molecule is involved in an intramolecular hydrogen bond [O···O 2.518 (6) and 2.547 (6) Å].

## Comment

Aversin, (I), is a metabolite isolated from a variant strain of *Aspergillus versicolor*. The structure derived previously from spectroscopic data (Bullock, Kirkaldy, Roberts & Underwood, 1963) has now been confirmed, except for the interchange of the OH and an OMe group. The two molecules in the asymmetric unit (Fig. 1) have virtually identical geometries and dimensions. The three six-membered rings of the anthraquinone skeleton and the attached five-membered ring are almost coplanar, with the final *cis*-fused five-membered ring displaced from this plane. This final five-membered ring has an envelope conformation, with C17 and C37 displaced from the respective plane of the other four atoms.

