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.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Fuyita, T., Iwasa, J. & Hansch, C. (1964). *J. Am. Chem. Soc.* **86**, 5175–5180.
Goeta, A. E. (1995). Doctoral thesis, Universidad Nacional de La Plata, Argentina.
Goeta, A. E., Punte, G. & Rivero, B. E. (1993). *Acta Cryst.* C**49**, 1996–1998.
Grand, C. A., Bruno Blanch, L., de Vázquez, A. G., Lufrano, P. & Baldini, O. A. N. (1984). *Acta Farm. Bonaerense*, **3**, 39–45.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Sheldrick, G. M. (1996). *SADABS. Empirical Absorption Correction Program*. University of Göttingen, Germany.
Siemens (1995). *SHELXTL*. Version 5.03/VMS. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1996*a*). *SMART. Data Collection Software*. Version 4.05. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1996*b*). *SAINT. Data Reduction Software*. Version 4.05. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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endo-1,4,4-Tribromo-3-methyltricyclo-[5.2.1.$0^{2,6}$]dec-8-ene-5,10-dione 10-Ethylene Acetal

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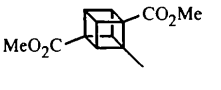
Abstract

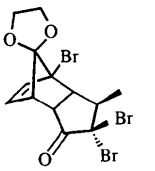
The molecular geometry of the title compound, $C_{13}H_{13}Br_3O_3$, is similar to that observed for the 3-chloro- analogue, *endo*-1,4,4-tribromo-3-methyltricyclo[5.2.1.$0^{2,6}$]dec-8-ene-5,10-dione 10-ethylene acetal.

Comment

We have recently described the preparation of dimethyl 2-methylcubane-1,4-dicarboxylate, (I) (Lowe *et al.*, 1994), through an indirect approach, which featured the *gem*-dibromoketone (II). The structure of (II) was originally assigned on the basis of NOE difference measurements and the X-ray structural analysis reported here was performed to confirm the assignment.



(I)



(II)

The molecular geometry is similar to that observed for the 3-chloro- analogue, *endo*-1,4,4-tribromo-3-chlorotricyclo[5.2.1.$0^{2,6}$]dec-8-ene-5,10-dione 10-ethylene ketal (Gable, Parker & Tsanaktsidis, 1994).

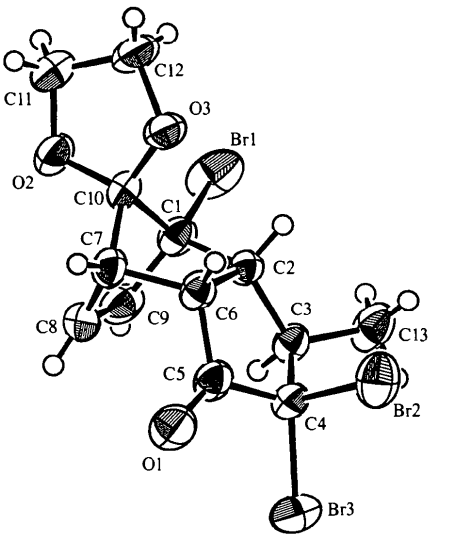


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of (II). Displacement ellipsoids are drawn at the 50% probability level. H atoms are given arbitrary radii of 0.1 Å.

Experimental

Compound (II) was prepared as described by Lowe *et al.* (1994) and crystals were obtained by slow evaporation from diethyl ether.

Crystal data

$C_{13}H_{13}Br_3O_3$
M_r = 456.96
Triclinic
$P\bar{1}$
a = 6.6161 (13) Å
b = 9.633 (2) Å
c = 12.692 (3) Å
α = 98.27 (2)°
β = 104.84 (2)°
γ = 106.74 (2)°
V = 727.8 (3) Å^3
Z = 2
D_x = 2.085 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 11.3–21.2°
μ = 8.314 mm^{-1}
T = 293 (1) K
Block
0.40 × 0.37 × 0.37 mm
Colourless

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Data collection

Enraf–Nonius CAD-4-MachS diffractometer
$\omega/2\theta$ scans
Absorption correction: Gaussian by integration (Sheldrick, 1976)
T_{min} = 0.041, T_{max} = 0.166
4197 measured reflections
3332 independent reflections
2462 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 27.47°
$h = -1 \rightarrow 8$
$k = -12 \rightarrow 12$
$l = -16 \rightarrow 16$
3 standard reflections
frequency: 160 min
intensity decay: 18%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.119
S = 1.055
3332 reflections
208 parameters
Methine H atoms refined, others riding
$w = 1/[\sigma^2(F_o^2) + (0.0563P)^2 + 1.0706P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.790 e Å^{-3}
$\Delta\rho_{min}$ = −0.667 e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0116 (15)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Br1—C1	1.927 (5)	C2—C3	1.541 (6)
Br2—C4	1.952 (5)	C3—C4	1.522 (6)
Br3—C4	1.921 (5)	C4—C5	1.529 (6)
C1—C2	1.545 (6)	C7—C10	1.551 (7)
C1—C10	1.557 (6)	C8—C9	1.308 (9)
C9—C1—C2	109.2 (4)	C3—C4—C5	105.4 (3)
C9—C1—C10	99.8 (4)	C3—C4—Br3	112.7 (3)
C2—C1—C10	100.7 (3)	C5—C4—Br3	112.9 (3)
C9—C1—Br1	115.8 (4)	C3—C4—Br2	113.1 (3)
C2—C1—Br1	113.5 (3)	C5—C4—Br2	104.0 (3)
C10—C1—Br1	116.0 (3)	Br3—C4—Br2	108.5 (2)
C13—C3—C4	116.8 (4)	C7—C10—C1	92.1 (3)
C13—C3—C2	115.0 (4)		

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1187). Services for accessing these data are described at the back of the journal.

References

Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Gable, R. W., Parker, K. J. & Tsanaktsidis, J. (1994). *Aust. J. Chem.* **47**, 963–968.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Lowe, D. A., Moorhouse, C. J., Walter, J. M. & Tsanaktsidis, J. (1994). *Aust. J. Chem.* **47**, 1647–1650.

Sheldrick, G. M. (1976). *SHELX*76. *Program for Crystal Structure Determination*. University of Cambridge, England.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

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Spirapril Hydrochloride Hydrate

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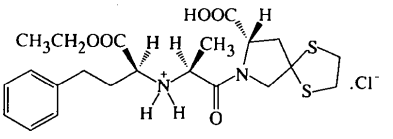
Abstract

The crystal structure of the monohydrate form of spirapril hydrochloride, (8*S*-{7[*R**(*R**)],8*R**})-7-(2-{[1-(ethoxycarbonyl)-3-phenylpropyl]amino}-1-oxopropyl)-1,4-dithia-7-azaspiro[4.4]nonane-8-carboxylic acid hydrochloride, $C_{22}H_{31}N_2O_5S_2^+.Cl^-.H_2O$, was determined. The spirapril molecules adopt a *trans* conformation along the amide bond. The incorporated water molecules form hydrogen bonds with the host molecules.

Comment

Spirapril hydrochloride, (I), belongs to a class of *N*-carboxyalkyl dipeptide ACE inhibitors. Like many other of these dipeptide drugs, it is not very stable in the solid state. The major degradation pathway involves an intramolecular cyclization reaction (Strickley, Visor, Lin & Gu, 1989; Gu, Strickley, Chi & Chowhan, 1990). In our study of the chemical stability of pharmaceutical solids, we chose spirapril hydrochloride as a model compound to investigate the effect of molecular mobility on solid-state reactivity. The structure of spirapril hydrochloride monohydrate was determined and the information on the molecular geometry will aid in the understanding of the molecular details of the cyclization reaction.



(I)

An *ORTEP*II (Johnson, 1976) drawing of spirapril hydrochloride is presented in Fig. 1. For the sake of simplicity, the protonated spirapril moiety is referred to as the cation. It adopts a *trans* conformation at the amide bond since C5 and C8 (higher priority than C11) are on the opposite side of the amide bond and the torsion angle between C8—N7 and C6—C5 is −175.7 (3)°. The same kind of *trans* conformation is also observed in other *N*-carboxyalkyl dipeptide ACE inhibitors such as quinapril hydrochloride (Hausin & Codding, 1991) and enalapril maleate (Precigoux, Geoffre & Leroy,