C1	0.6766 (3)	0.7342 (2)	0.04868 (6)	0.0240 (6)
C2	0.6738 (3)	0.6729 (2)	0.01080 (6)	0.0255 (7)
C3	0.5507 (3)	0.5207 (3)	0.08034 (7)	0.0241 (6)
C4	0.4240 (3)	0.5193 (3)	0.07648 (8)	0.0364 (8)

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

N—C1	1.518 (3)	C1—C2	1.514 (3)
N—C3	1.514 (3)	C3—C4	1.519 (4)
C1—N—C1^i	107.3 (3)	C3—N—C3^i	106.3 (3)
C1—N—C3	110.71 (15)	C2—C1—N	115.0 (2)
C1—N—C3^i	110.92 (15)	N—C3—C4	113.9 (2)

Symmetry code: (i) $\frac{4}{3} - x, \frac{2}{3} - x + y, \frac{1}{6} - z$.

All non-H atoms were located from direct methods. H atoms were included in the calculations as riding atoms with common isotropic displacement parameters. Methyl H atoms were allowed to rotate about C—C bonds. Reflections with l odd are systematically weak, giving an apparent pseudo-cell with c = 17.58 Å.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1993). Cell refinement: *CAD-4-PC* Software. Data reduction: *XCAD4* (Harms, 1995). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1971) and *SHELXTL* (Siemens, 1995).

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: BK1293). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Cheng, J., Xenopoulos, A. & Wunderlich, B. (1992*a*). *Mol. Cryst. Liq. Cryst.* **220**, 105–125.
Cheng, J., Xenopoulos, A. & Wunderlich, B. (1992*b*). *Mol. Cryst. Liq. Cryst.* **220**, 127–157.
Cheng, J., Xenopoulos, A. & Wunderlich, B. (1993). *Mol. Cryst. Liq. Cryst.* **225**, 337–353.
Enraf–Nonius (1993). *CAD-4-PC Software*. Version 1.2. Enraf–Nonius, Delft, The Netherlands.
Harms, K. (1995). *XCAD4. Program for the Lp Correction of Enraf–Nonius Four-Circle Diffractometer Data*. University of Marburg, Germany.
Johnson, C. K. (1971). *ORTEPII*. Report ORNL-3794, revised. Oak Ridge National Laboratory, Tennessee, USA.
Ralle, M., Xenopoulos, A., Habenschuss, A. & Wunderlich, B. (1996). In preparation.
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.
Siemens (1995). *SHELXTL*. Version 5/*IRIX*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Vincent, B. V., Knop, O., Linden, A., Cameron, T. S. & Robertson, K. N. (1988). *Can. J. Chem.* **66**, 3060–3069.
Xenopoulos, A., Cheng, J. & Wunderlich, B. (1993). *Mol. Cryst. Liq. Cryst.* **226**, 87–102.
Xenopoulos, A., Cheng, J., Yasuniva, M. & Wunderlich, B. (1992). *Mol. Cryst. Liq. Cryst.* **214**, 63–79.
Xenopoulos, A., Ralle, M., Habenschuss, A. & Wunderlich, B. (1996). *Powder Diffr.* **11**, 246–249.

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Ethyl *N*-Methyl-2-pyrrolylcarbonyl-hydrazinocarboxylate

MALVA LIU-GONZALEZ,[a] FRANCISCO SANZ-RUIZ,[a] MARIA EUGENIA GONZALEZ-ROSENDE,[b] OSCAR LOZANO-LUCÍA,[b] ELENA ZABALLOS-GARCÍA[b] AND JOSE SEPULVEDA-ARQUÉS[b]

[a]*Departamento de Termología, Facultad de Física, Universidad de Valencia, Dr. Moliner 50, 47100 Burjassot (Valencia), Spain, and* [b]*Departamento de Química Orgánica, Facultad de Farmacia, Universidad de Valencia, Avda. Vte. Andrés Estelles s/n, 47100 Burjassot (Valencia), Spain. E-mail: liu@evalrx.scsie.uv.es*

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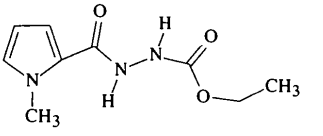
Abstract

The title compound, $C_9H_{13}N_3O_3$, consists of an *N*-methyl-substituted pyrrolylcarbonyl moiety bonded at position 2 to the N′ atom of a hydrazino chain.

Comment

The C—C and C—N interatomic distances in the title compound, (I), are similar to those found in other pyrrole structures (Cullen, Pepe, Meyer, Falk & Grubmayr, 1979; Fritz, Henlin, Reisen, Tschamber & Zehnderand, 1988; Ruben, Bates, Zalkin & Templeton, 1974).



(I)

The C2—O1 bond [1.232 (2) Å], a urea 'vinylogue', is slightly longer than C3—O2 [1.223(2) Å] in the carbamate group. The C2—N2 interatomic distance is also longer than N3—C3. The torsion angle O1—C2—C2′—N1′ of −8.4 (2)° (Table 1) indicates that the carbonyl group has an *s-cis* disposition with respect to the C—N bond of the pyrrole ring. The relative disposition of the pyrrolylcarbonyl and ethoxycarbonyl substituents on the hydrazine chain afford a torsion

angle C3—N3—N2—C2 of −79.6 (2)°. A perspective view of the title molecule and the arbitrary atom-labelling scheme are shown in Fig. 1.

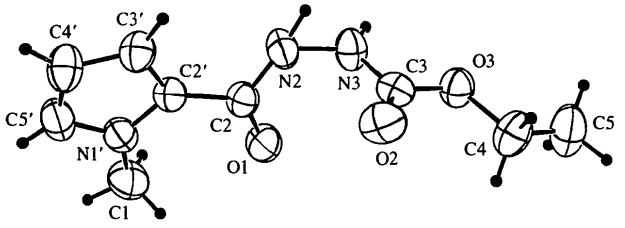


Fig. 1. View of $C_9H_{13}N_3O_3$ showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as small circles of arbitrary radii.

Experimental

In the course of a study on the reactivity of 2-formylpyrroles with azodicarbonyl compounds, we found that the reaction of 1-methyl-2-formylpyrrole with diethyl azodicarboxylate in CH_2Cl_2 at 413 K in an enclosed steel reactor gives rise to the title compound with a yield of 17%. The structure inferred from elemental analysis and spectral data was confirmed by X-ray analysis and compared with the examples reported in the literature for this kind of compound generated *via* radical intermediates (Alder & Noble, 1943; Huisgen & Jacob, 1954).

Crystal data

$C_9H_{13}N_3O_3$
M_r = 211.22
Trigonal
$P3_2$
a = 9.363 (1) Å
c = 11.015 (2) Å
V = 836.2 (3) Å^3
Z = 3
D_x = 1.258 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 6–10°
μ = 0.09 mm^{-1}
T = 293 (2) K
Cylindrical
0.25 × 0.18 × 0.18 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
1844 measured reflections
1844 independent reflections
1496 reflections with $I > 3\sigma(I)$

θ_{max} = 24.97°
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 11$
$l = -13 \rightarrow 13$
3 standard reflections
every 200 reflections
frequency: 60 min
intensity decay: 2.4%

Refinement

Refinement on F
R = 0.036
wR = 0.046
S = 1.734
1496 reflections
174 parameters
H-atom coordinates refined
$w = 1/\sigma(F)^2$
$(\Delta/\sigma)_{max}$ = 0.060

$\Delta\rho_{max}$ = 0.333 e Å^{-3}
$\Delta\rho_{min}$ = −0.137 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Flack parameter for absolute configuration determination = −0.1871

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

C2—C2′	1.464 (2)	C2′—C3′	1.385 (2)
C2—O1	1.232 (2)	N1′—C5′	1.357 (2)
C2—N2	1.354 (2)	N1′—C1	1.462 (3)
N2—N3	1.377 (2)	C2′—N1′	1.379 (2)
C3′—C4′	1.396 (2)	C3—O2	1.223 (2)
O3—C3	1.330 (2)	C4—C5	1.497 (3)
O3—C4	1.456 (3)	N3—C3	1.349 (3)
C5′—C4′	1.365 (3)		
C5′—N1′—C1	123.1 (2)	O3—C4—C5	106.8 (2)
C2′—N1′—C1	128.4 (1)	C3—O3—C4	115.8 (1)
C2′—N1′—C5′	108.2 (1)	N3—C3—O2	125.1 (1)
C3′—C4′—C5′	107.4 (2)	O1—C2—N2	122.1 (1)
N1′—C5′—C4′	109.3 (1)	O3—C3—O2	125.2 (2)
N1′—C2′—C3′	107.7 (1)	C2′—C2—O1	123.7 (1)
C2—N2—N3	120.4 (2)	C2′—C2—N2	114.2 (1)
C2—C2′—C3′	128.9 (1)	C3—N3—N2	119.9 (1)
C2′—C3′—C4′	107.3 (2)	O3—C3—N3	109.7 (1)
C2—C2′—N1′	123.3 (1)		
O1—C2—C2′—N1′	−8.4 (2)	C3—O3—C4—C5	170.9 (2)
O1—C2—C2′—C3′	169.5 (2)	C2—C2′—N1′—C5′	178.7 (2)
N2—C2—C2′—N1′	170.9 (2)	C2—C2′—N1′—C1	−7.0 (3)
N2—C2—C2′—C3′	−11.2 (3)	C2—C2′—C3′—C4′	−178.3 (2)
C2′—C2—N2—N3	174.5 (1)	C1—N1′—C5′—C4′	−175.2 (2)
O1—C2—N2—N3	−6.2 (2)	N2—N3—C3—O3	173.9 (1)
C4—O3—C3—N3	−175.9 (1)	N2—N3—C3—O2	−7.4 (2)
C4—O3—C3—O2	5.4 (2)	C3—N3—N2—C2	−79.6 (2)

Data collection: *CAD-4 Software* (Enraf–Nonius, 1991). Data reduction: *PROCESS* in *MolEN* (Fair, 1990). Program(s) used to solve structure: *MULTAN*11/82 (Main *et al.*, 1982). Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEP* (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93 (Sheldrick, 1993).

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: KA1194). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Alder, K. & Noble, T. (1943). *Ber. Dtsch Chem. Ges.* **76**, 54–57.
Cullen, D. L., Pepe, G., Meyer, E. F., Falk, H. & Grubmayr, H. (1979). *J. Chem. Soc. Perkin Trans.* 2, pp. 999–1004.
Enraf–Nonius (1991). *CAD-4 Software*. Enraf–Nonius, Delft, The Netherlands.
Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
Fritz, H., Henlin, J. M., Reisen, A., Tschamber, T. & Zehnderand, M. (1988). *J. Helv. Chim. Acta*, **71**, 822–834.
Huisgen, R. & Jacob, F. (1954). *Liebigs Ann. Chem.* **590**, 37–54.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Universities of York, England, and Louvain, Belgium.
Ruben, H., Bates, H., Zalkin, A. & Templeton, D. H. (1974). *Acta Cryst.* B**30**, 1631–1633.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.