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3-Ethoxycarbonyl-2,2,5,5-tetramethyl-1*H*-2,5-dihydropyrrol-1-yloxyl

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.044
wR factor = 0.152
Data-to-parameter ratio = 11.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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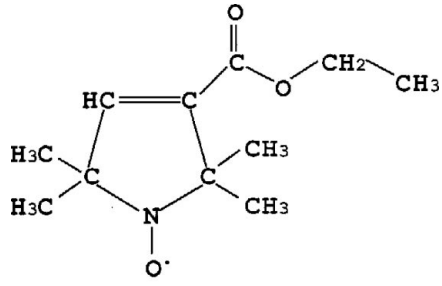
The crystal and molecular structure of the title compound, $C_{11}H_{18}NO_3$, has been determined from X-ray diffraction data. The compound crystallizes in the centrosymmetric space group *Pnma*. The main part of the molecule lies at a special position on the mirror plane.

Comment

The title molecule, (I), and the labeling scheme are shown in Fig. 1. As expected, the part of the molecule containing the pyrrol ring with the ethoxycarbonyl group is planar with standard bond distances and angles similar to that in hexa-2,4-diyne-6-(2,2,5,5-tetramethyl-1-oxyl-3-pyrrolin-3-carboxylate)-1-ol (Wiley, 1991). Two atoms, C12 and O6, showed higher values of atomic displacement parameters perpendicular to the plane of molecule, *i.e.* ellipsoids are elongated along the *b*-axis direction. In contrast (Wiley, 1991), there are no hydrogen bonds because of a lack of donor atoms. The packing diagram of the structure is presented in Fig. 2.



(I)

Experimental

A solution of 3-carboxy-2,2,5,5-tetramethyl-1*H*-2,5-dihydropyrrol-1-yloxyl (1.84 g, 0.01 mol), dicyclohexylcarbodiimide (2.1 g, 0.01 mol), ethanol (1 g, 0.022 mol) and 4-(pyrrol-1-yl)pyridine (0.1 g) in 20 ml dichloromethane was stirred overnight. After filtering off the dicyclohexylurea, the solution was extracted with 5% solution of sodium hydrogencarbonate and dried with magnesium sulfate. The methylene chloride was evaporated and the crude product was crystallized from hexane solution.

Crystal data

$C_{11}H_{18}NO_3$
M_r = 212.26
Orthorhombic, *Pnma*
a = 10.4710 (4) Å
b = 9.3700 (3) Å
c = 12.3500 (4) Å
V = 1211.70 (7) Å^3
Z = 4
D_x = 1.164 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 12475 reflections
θ = 1.0–27.5°
μ = 0.08 mm^{-1}
T = 150 K
Prism, yellow
0.4 × 0.3 × 0.2 mm

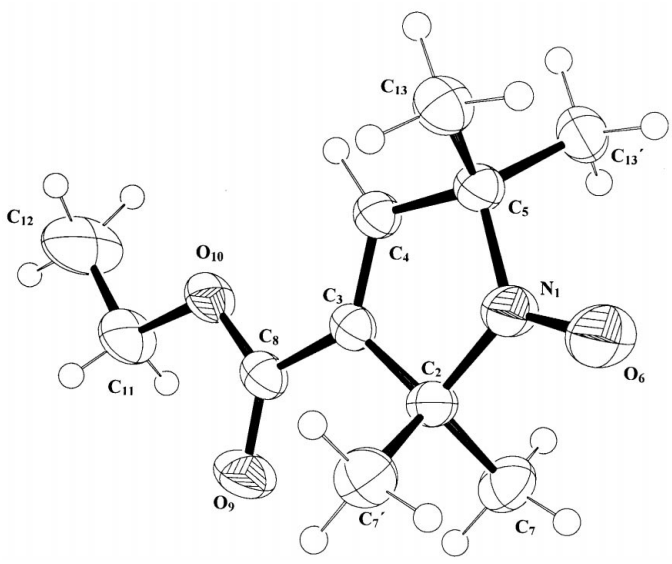


Figure 1
The structure of the title compound showing the atom-numbering scheme. Displacement ellipsoids of the non-H atoms are drawn at the 50% probability level.

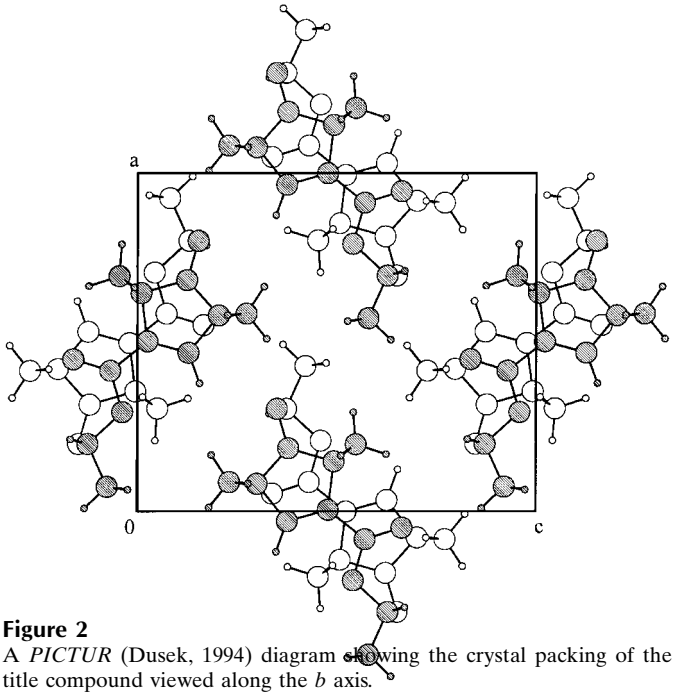


Figure 2
A *PICTUR* (Dusek, 1994) diagram showing the crystal packing of the title compound viewed along the *b* axis.

Data collection

Nonius KappaCCD area-detector diffractometer	$R_{int} = 0.028$
φ and ω scans	$\theta_{max} = 27.5^\circ$
15 701 measured reflections	$h = -13 \rightarrow 13$
1464 independent reflections	$k = -12 \rightarrow 12$
1216 reflections with $I > 2\sigma(I)$	$l = -15 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.044$	$(\Delta/\sigma)_{max} = 0.009$
$wR(F^2) = 0.152$	$\Delta\rho_{max} = 0.27$ e Å^{-3}
$S = 1.22$	$\Delta\rho_{min} = -0.15$ e Å^{-3}
1462 reflections	Extinction correction: *SHELXL*97
123 parameters	Extinction coefficient: 0.00003 (1)
H atoms: see below	

Table 1
Selected geometric parameters (Å, °).

N1—O6	1.270 (2)	C4—C5	1.496 (2)
N1—C5	1.477 (2)	C5—C13	1.526 (1)
N1—C2	1.476 (2)	C8—O9	1.203 (2)
C2—C3	1.506 (2)	C8—O10	1.331 (2)
C2—C7	1.529 (1)	O10—C11	1.458 (2)
C3—C4	1.331 (2)	C11—C12	1.456 (3)
C3—C8	1.478 (2)		
O6—N1—C5	122.5 (1)	N1—C5—C4	99.7 (1)
C5—N1—C2	115.2 (1)	N1—C5—C13	110.53 (8)
N1—C2—C3	99.4 (1)	C4—C5—C13	112.42 (8)
N1—C2—C7	109.58 (9)	C13^{i}—C5—C13	110.7 (1)
C3—C2—C7	113.66 (8)	O9—C8—O10	123.7 (1)
C7—C2—C7^{i}	110.3 (1)	O9—C8—C3	123.6 (1)
C4—C3—C8	125.4 (1)	O10—C8—C3	112.6 (1)
C4—C3—C2	112.7 (1)	C8—O10—C11	116.4 (1)
C3—C4—C5	112.9 (1)	O10—C11—C12	108.5 (2)

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

Two reflections 011 and 101 were omitted from the final refinement due to the large beam stop used. The systematic absences showed two possible space groups, non-centrosymmetric $Pn2_1a$ and centrosymmetric *Pnma*. The phase problem was solved in the space group *Pnma*. All H atoms were found in the difference Fourier maps and freely refined with isotropic displacement parameters. The refinement in non-centrosymmetric $Pn2_1a$ led to lowering the *R* value from 0.044 to 0.042. However, the refinement in this space group was less stable and led to unrealistic positions of H atoms. Free refinement in *Pnma* gave C—H bond distances in the interval 0.96–1.03 Å and free refinement in $Pn2_1a$ gave deviations of non-H atoms from planarity less than 2 s.u.'s and C—H bonds in the range 0.85–1.35 Å. Therefore, the *Pnma* group was prefered.

Data collection, cell refinement and data reduction: *COLLECT* (Hooft, 1998) and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97 and *ORTEP*-3.

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