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2-*tert*-Butyl 4-ethyl 3,5-dimethyl-1*H*-pyrrole-2,4-dicarboxylate

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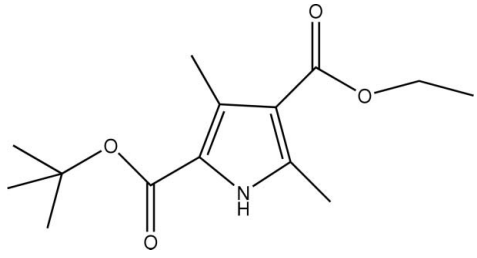
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.075; wR factor = 0.177; data-to-parameter ratio = 14.0.

In the title compound, $C_{14}H_{21}NO_4$, all the non-H atoms, except for one methyl C atom, lie on a crystallographic mirror plane. An N—H···O hydrogen bond helps to consolidate the crystal packing. A short intramolecular C—H···O contact also occurs.

Related literature

For background, see: Sun *et al.* (2002).



Experimental

Crystal data

$C_{14}H_{21}NO_4$
M_r = 267.32
Monoclinic, $C2/m$
a = 18.570 (4) Å
b = 7.1420 (14) Å
c = 12.431 (3) Å
β = 116.46 (3)°
V = 1476.0 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.40 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.966, T_{max} = 0.991
1578 measured reflections
1578 independent reflections
920 reflections with $I > 2\sigma(I)$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.075
$wR(F^2)$ = 0.177
S = 1.11
1578 reflections
113 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^{i}	0.86	2.08	2.928 (5)	169
C9—H9A···O2	0.96	2.25	2.965 (7)	131

Symmetry code: (i) $-x, y, -z$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2539).

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supplementary materials

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2-*tert*-Butyl 4-ethyl 3,5-dimethyl-1*H*-pyrrole-2,4-dicarboxylate

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Comment

As part of our owning studies of pyrrole derivatives (Sun *et al.*, 2002), we report here the crystal structure of the title compound, (I). All the non-hydrogen atoms except C12 lie on a crystallographic mirror plane (Fig. 1).

An N—H···O hydrogen bond (Table 1) helps to establish the crystal packing in (I). A short intramolecular C—H···O contact also occurs, based on the geometrically positioned H9A atom, which lies on the mirror plane.

Experimental

tert-Butyl-3-oxobutyrate (20 mmol) in acetic acid (4 ml) was cooled in an ice bath to about 278 K. Sodium nitrite (20 mmol) was added over 45 minutes keeping the temperature under 288 K. The mixture was stirred for 30 minutes and then left standing for 3 h, yielding *tert*-buty-2-hydroximino-3-oxobutyarate. Ethyl-3-oxobutyrate (20 mmol), zinc dust (50 g) and the crude *tert*-buty-2-hydroximino-3-oxobutyarate in acetic acid (6 ml) were stirred at 343 K for 1 hr·The reaction mixture was poured into 200 ml water and the filtrate was collected to obtain the title compound. Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a methanol solution.

Refinement

The H atoms were geometrically placed (C—H = 0.96 Å N—H = 0.86 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C,*N*) or 1.5U_{eq}(methyl C).

Figures

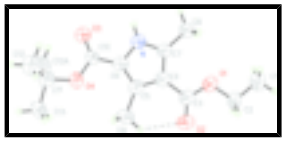

Fig. 1. The molecular structure of (I) with displacement ellipsoids drawn at the 30% probability level (arbitrary spheres for the H atoms). The intramolecular C9—H9A···O2 contact is indicated by a dashed line. C12A is generated by the symmetry operation (*x*, −*y*, *z*).

2-*tert*-Butyl 4-ethyl 3,5-dimethyl-1*H*-pyrrole-2,4-dicarboxylate

Crystal data

$C_{14}H_{21}NO_4$	F_{000} = 576
M_r = 267.32	D_x = 1.203 Mg m^{-3}
Monoclinic, *C*2/*m*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2y	Cell parameters from 25 reflections
a = 18.570 (4) Å	θ = 9–13°

b = 7.1420 (14) Å	μ = 0.09 mm^{-1}
c = 12.431 (3) Å	T = 293 (2) K
β = 116.46 (3)º	Block, white
V = 1476.0 (5) Å^3	0.40 × 0.10 × 0.10 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.0000
Radiation source: fine-focus sealed tube	θ_{max} = 26.0º
Monochromator: graphite	θ_{min} = 1.8º
T = 293(2) K	h = −22→20
ω/2θ scans	k = 0→8
Absorption correction: ψ scan (North *et al.*, 1968)	l = 0→15
T_{min} = 0.966, T_{max} = 0.991	3 standard reflections
1578 measured reflections	every 200 reflections
1578 independent reflections	intensity decay: none
920 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.075	H-atom parameters constrained
$wR(F^2)$ = 0.177	$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 2P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.11	$(\Delta/\sigma)_{max}$ = 0.029
1578 reflections	$\Delta\rho_{max}$ = 0.30 e Å^{-3}
113 parameters	$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.1275 (2)	0.0000	0.0227 (3)	0.0429 (9)
H1	0.0763	0.0000	−0.0214	0.051*
O1	0.32742 (17)	0.0000	−0.0354 (3)	0.0565 (9)
O2	0.39992 (18)	0.0000	0.1619 (3)	0.0745 (12)
O3	0.04155 (18)	0.0000	0.1558 (3)	0.0615 (10)
O4	0.15571 (17)	0.0000	0.3287 (2)	0.0527 (9)
C1	0.3784 (3)	0.0000	−0.1789 (4)	0.0621 (14)
H1A	0.4260	0.0000	−0.1911	0.093*
H1B	0.3471	−0.1098	−0.2152	0.093*
C2	0.4002 (2)	0.0000	−0.0504 (4)	0.0473 (11)
H2A	0.4323	−0.1087	−0.0142	0.057*
C3	0.3360 (3)	0.0000	0.0753 (4)	0.0480 (11)
C4	0.2571 (2)	0.0000	0.0810 (4)	0.0449 (11)
C5	0.2468 (2)	0.0000	0.1835 (4)	0.0487 (11)
C6	0.1645 (2)	0.0000	0.1481 (4)	0.0430 (10)
C7	0.1821 (2)	0.0000	−0.0197 (4)	0.0420 (10)
C8	0.1544 (3)	0.0000	−0.1516 (4)	0.0532 (12)
H8A	0.0969	0.0000	−0.1966	0.080*
H8B	0.1760	0.1098	−0.1712	0.080*
C9	0.3123 (3)	0.0000	0.3113 (4)	0.0628 (14)
H9A	0.3645	0.0000	0.3130	0.094*
H9B	0.3066	0.1098	0.3514	0.094*
C10	0.1142 (3)	0.0000	0.2074 (4)	0.0475 (12)
C12	0.0679 (2)	0.1808 (6)	0.3897 (3)	0.0838 (13)
H12A	0.1018	0.2865	0.3976	0.126*
H12B	0.0483	0.1882	0.4492	0.126*
H12C	0.0232	0.1809	0.3110	0.126*
C13	0.1879 (3)	0.0000	0.5340 (4)	0.0783 (18)
H13A	0.2211	−0.1066	0.5408	0.117*
H13C	0.1698	0.0000	0.5952	0.117*
C11	0.1162 (3)	0.0000	0.4075 (4)	0.0581 (13)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0415 (18)	0.042 (2)	0.0424 (19)	0.000	0.0165 (16)	0.000
O1	0.0466 (17)	0.060 (2)	0.065 (2)	0.000	0.0271 (16)	0.000
O2	0.0435 (18)	0.110 (3)	0.058 (2)	0.000	0.0110 (16)	0.000
O3	0.0490 (18)	0.079 (3)	0.0529 (18)	0.000	0.0199 (15)	0.000
O4	0.0554 (18)	0.062 (2)	0.0473 (17)	0.000	0.0288 (15)	0.000
C1	0.075 (3)	0.044 (3)	0.075 (3)	0.000	0.041 (3)	0.000
C2	0.052 (2)	0.034 (3)	0.063 (3)	0.000	0.032 (2)	0.000
C3	0.049 (3)	0.038 (3)	0.057 (3)	0.000	0.023 (2)	0.000
C4	0.047 (2)	0.037 (3)	0.052 (3)	0.000	0.023 (2)	0.000

C5	0.044 (2)	0.046 (3)	0.053 (3)	0.000	0.018 (2)	0.000
C6	0.045 (2)	0.028 (3)	0.049 (2)	0.000	0.015 (2)	0.000
C7	0.046 (2)	0.032 (3)	0.044 (2)	0.000	0.017 (2)	0.000
C8	0.056 (3)	0.050 (3)	0.055 (3)	0.000	0.025 (2)	0.000
C9	0.062 (3)	0.064 (4)	0.051 (3)	0.000	0.016 (2)	0.000
C10	0.045 (2)	0.054 (3)	0.039 (2)	0.000	0.014 (2)	0.000
C12	0.085 (3)	0.087 (3)	0.083 (3)	0.008 (3)	0.041 (2)	−0.006 (3)
C13	0.085 (4)	0.098 (5)	0.050 (3)	0.000	0.028 (3)	0.000
C11	0.059 (3)	0.057 (4)	0.056 (3)	0.000	0.024 (2)	0.000

Geometric parameters (Å, °)

N1—C7	1.335 (5)	C5—C6	1.389 (6)
N1—C6	1.395 (5)	C5—C9	1.511 (6)
N1—H1	0.8600	C6—C10	1.425 (6)
O1—C3	1.312 (5)	C7—C8	1.483 (5)
O1—C2	1.444 (5)	C8—H8A	0.9601
O2—C3	1.195 (5)	C8—H8B	0.9600
O3—C10	1.208 (5)	C9—H9A	0.9600
O4—C10	1.353 (5)	C9—H9B	0.9600
O4—C11	1.463 (5)	C12—C11	1.531 (5)
C1—C2	1.464 (6)	C12—H12A	0.9600
C1—H1A	0.9600	C12—H12B	0.9600
C1—H1B	0.9601	C12—H12C	0.9600
C2—H2A	0.9600	C13—C11	1.542 (6)
C3—C4	1.500 (6)	C13—H13A	0.9601
C4—C5	1.370 (6)	C13—H13C	0.9589
C4—C7	1.397 (5)	C11—C12^{i}	1.531 (5)
C7—N1—C6	111.0 (3)	C4—C7—C8	135.0 (4)
C7—N1—H1	124.5	C7—C8—H8A	113.1
C6—N1—H1	124.5	C7—C8—H8B	107.7
C3—O1—C2	116.8 (3)	H8A—C8—H8B	109.5
C10—O4—C11	122.7 (3)	C5—C9—H9A	110.8
C2—C1—H1A	110.2	C5—C9—H9B	108.8
C2—C1—H1B	109.1	H9A—C9—H9B	109.5
H1A—C1—H1B	109.5	O3—C10—O4	122.6 (4)
O1—C2—C1	108.8 (4)	O3—C10—C6	124.0 (4)
O1—C2—H2A	111.2	O4—C10—C6	113.4 (4)
C1—C2—H2A	108.8	C11—C12—H12A	109.5
O2—C3—O1	123.5 (4)	C11—C12—H12B	109.5
O2—C3—C4	123.8 (4)	H12A—C12—H12B	109.5
O1—C3—C4	112.6 (4)	C11—C12—H12C	109.5
C5—C4—C7	109.7 (4)	H12A—C12—H12C	109.5
C5—C4—C3	126.0 (4)	H12B—C12—H12C	109.5
C7—C4—C3	124.2 (4)	C11—C13—H13A	108.6
C4—C5—C6	107.1 (4)	C11—C13—H13C	111.1
C4—C5—C9	126.7 (4)	H13A—C13—H13C	111.7
C6—C5—C9	126.2 (4)	O4—C11—C12^{i}	109.8 (3)

N1—C6—C5	106.2 (4)	O4—C11—C12	109.8 (3)
N1—C6—C10	117.9 (4)	C12^{i}—C11—C12	115.0 (4)
C5—C6—C10	135.9 (4)	O4—C11—C13	102.7 (4)
N1—C7—C4	105.9 (3)	C12^{i}—C11—C13	109.4 (3)
N1—C7—C8	119.1 (4)	C12—C11—C13	109.4 (3)
C3—O1—C2—C1	180.0	C9—C5—C6—C10	0.0
C2—O1—C3—O2	0.0	C6—N1—C7—C4	0.0
C2—O1—C3—C4	180.0	C6—N1—C7—C8	180.0
O2—C3—C4—C5	0.0	C5—C4—C7—N1	0.0
O1—C3—C4—C5	180.0	C3—C4—C7—N1	180.0
O2—C3—C4—C7	180.0	C5—C4—C7—C8	180.0
O1—C3—C4—C7	0.0	C3—C4—C7—C8	0.0
C7—C4—C5—C6	0.0	C11—O4—C10—O3	0.0
C3—C4—C5—C6	180.0	C11—O4—C10—C6	180.0
C7—C4—C5—C9	180.0	N1—C6—C10—O3	0.0
C3—C4—C5—C9	0.0	C5—C6—C10—O3	180.0
C7—N1—C6—C5	0.0	N1—C6—C10—O4	180.0
C7—N1—C6—C10	180.0	C5—C6—C10—O4	0.0
C4—C5—C6—N1	0.0	C10—O4—C11—C12^{i}	63.7 (3)
C9—C5—C6—N1	180.0	C10—O4—C11—C12	−63.7 (3)
C4—C5—C6—C10	180.0	C10—O4—C11—C13	180.0

Symmetry codes: (i) *x*, −*y*, *z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3^{ii}	0.86	2.08	2.928 (5)	169
C9—H9A···O2	0.96	2.25	2.965 (7)	131

Symmetry codes: (ii) −*x*, *y*, −*z*.

Fig. 1