

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

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

Hong-Xin Cai* and Zhao-Po Zhang

Department of Physics and Chemistry, Henan Polytechnic University, Jiaozuo 454000, People's Republic of China

Correspondence e-mail: me2001@hpu.edu.cn

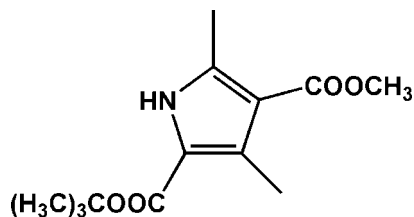
Received 8 April 2012; accepted 4 May 2012

 Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.071; wR factor = 0.250; data-to-parameter ratio = 15.1.

In the title molecule, $\text{C}_{13}\text{H}_{19}\text{NO}_4$, except for two C atoms of the *tert*-butyl group, the non-H atoms are almost coplanar (r.m.s. deviation = 0.2542 Å). In the crystal, molecules are linked into centrosymmetric dimers by two intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming an $R_2^2(10)$ ring motif.

Related literature

For complexes of Schiff bases containing a pyrrole unit, see: Wu *et al.* (2003); Wang *et al.* (2008). For the synthesis of the title compound, see: Sun *et al.* (2003).



Experimental

Crystal data

 $\text{C}_{13}\text{H}_{19}\text{NO}_4$
 $M_r = 253.29$

 Monoclinic, $P2_1/c$
 $a = 11.788$ (4) Å

 $b = 17.045$ (6) Å

 $c = 7.229$ (2) Å

 $\beta = 106.420$ (7)°

 $V = 1393.2$ (8) Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 0.09$ mm⁻¹
 $T = 296$ K

 $0.19 \times 0.18 \times 0.16$ mm

Data collection

Bruker SMART APEX CCD

diffractometer

Absorption correction: multi-scan

(SADABS; Bruker, 2007)

 $T_{\min} = 0.983$, $T_{\max} = 0.986$

7149 measured reflections

2458 independent reflections

 1334 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.050$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.071$
 $wR(F^2) = 0.250$
 $S = 1.02$

2458 reflections

163 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.28$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.21$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O4}^i$	0.86	2.14	2.974 (4)	165

 Symmetry code: (i) $-x + 2, -y, -z + 2$.

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

The authors are grateful for financial support from the Doctoral Foundation of Henan Polytechnic University (grant No. B2009-70 648364).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: VM2170).

References

- Bruker (2007). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Sun, L., *et al.* (2003). *J. Med. Chem.* **46**, 1116–1119.
- Wang, Y., Yang, Z.-Y. & Chen, Z.-N. (2008). *Bioorg. Med. Chem. Lett.* **18**, 298–303.
- Wu, Z. K., Chen, Q. Q., Xiong, S. X., Xin, B., Zhao, Z. W., Jiang, L. J. & Ma, J. S. (2003). *Angew. Chem. Int. Ed.* **42**, 3271–3274.

supporting information

Acta Cryst. (2012). E68, o1697 [doi:10.1107/S1600536812020120]

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

Hong-Xin Cai and Zhao-Po Zhang

S1. Comment

Schiff bases containing pyrrole units have been extensively investigated due to their excellent coordination abilities (Wu *et al.*, 2003; Wang *et al.*, 2008). As part of our studies on bis(pyrrol-2-yl-methyleneamine) ligands, the crystal structure of the title compound is reported here.

In the title molecule (Fig. 1), except for C12 and C13 atoms of the *tert*-butyl, the non-hydrogen atoms are almost coplanar (r.m.s. deviation of the non-hydrogen atoms being 0.2542 Å). In the crystal, the molecules are linked into centrosymmetric dimers by two intermolecular N—H···O hydrogen bonds (Table 1), forming a $R_2^2(10)$ ring motif (Fig. 2).

S2. Experimental

The 2-*tert*-butyl 4-methyl 3,5-dimethyl-1*H*-pyrrole-2,4-dicarboxylate was prepared by a Knorr-type reaction from the condensation of methyl acetoacetate and *tert*-butyl oximinoacetoacetate according to a literature method (Sun *et al.*, 2003).

S3. Refinement

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

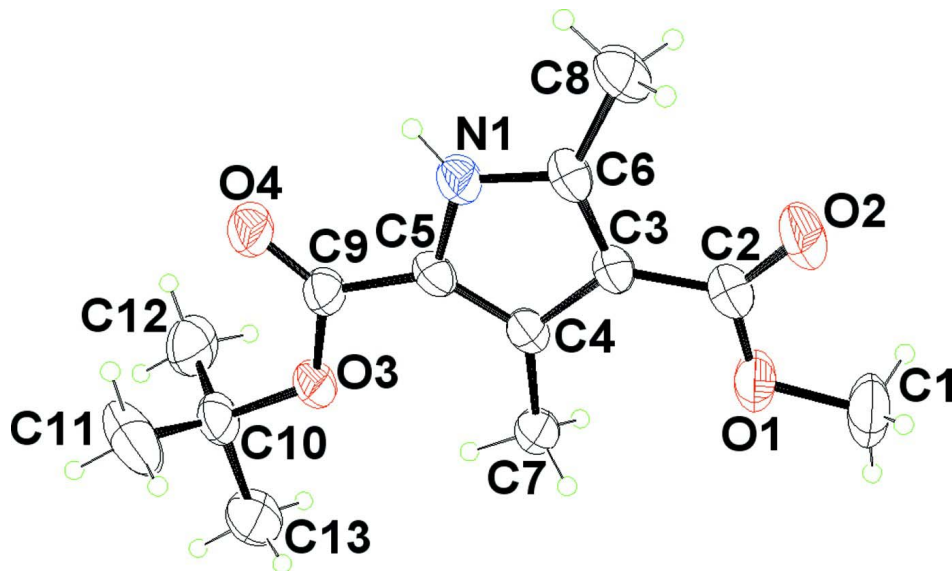
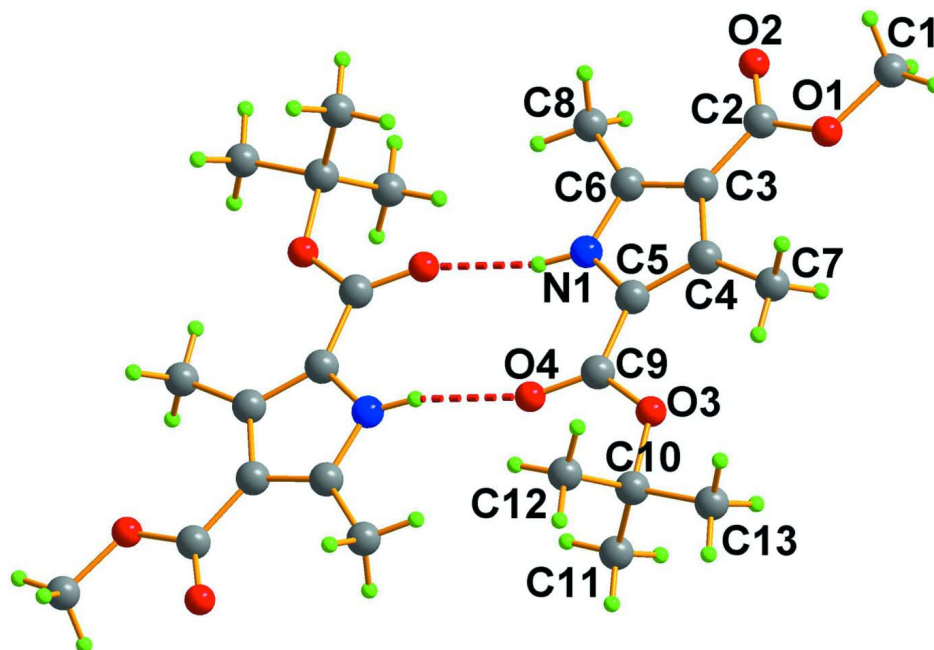


Figure 1

The molecular structure shown with 30% probability displacement ellipsoids.

**Figure 2**

The dimer of the title compounds formed *via* N—H···O hydrogen bonds shown as the dashed lines. Unlabelled atoms are related with the labelled ones by symmetry operation $(-x, 1/2 - y, 1/2 + z)$.

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

Crystal data

$C_{13}H_{19}NO_4$

$M_r = 253.29$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 11.788\ (4)\ \text{\AA}$

$b = 17.045\ (6)\ \text{\AA}$

$c = 7.229\ (2)\ \text{\AA}$

$\beta = 106.420\ (7)^\circ$

$V = 1393.2\ (8)\ \text{\AA}^3$

$Z = 4$

$F(000) = 544$

$D_x = 1.208\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1135 reflections

$\theta = 3.0\text{--}21.5^\circ$

$\mu = 0.09\ \text{mm}^{-1}$

$T = 296\ \text{K}$

Plate, colorless

$0.19 \times 0.18 \times 0.16\ \text{mm}$

Data collection

Bruker SMART CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2007)

$T_{\min} = 0.983$, $T_{\max} = 0.986$

7149 measured reflections

2458 independent reflections

1334 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.050$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 2.2^\circ$

$h = -13 \rightarrow 14$

$k = -19 \rightarrow 20$

$l = -8 \rightarrow 7$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.071$
 $wR(F^2) = 0.250$
 $S = 1.02$
 2458 reflections
 163 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.1445P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.28 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.21 \text{ e } \text{\AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	1.1539 (4)	0.4652 (3)	1.0373 (8)	0.1274 (19)
H1B	1.0961	0.5063	1.0193	0.191*
H1C	1.1953	0.4696	0.9409	0.191*
H1D	1.2091	0.4699	1.1630	0.191*
C2	1.1615 (3)	0.3246 (2)	1.0408 (5)	0.0768 (11)
C3	1.0936 (3)	0.25230 (19)	1.0199 (4)	0.0587 (9)
C4	0.9687 (3)	0.24090 (18)	0.9753 (4)	0.0554 (8)
C5	0.9521 (2)	0.16057 (19)	0.9724 (4)	0.0586 (9)
C6	1.1478 (3)	0.1795 (2)	1.0403 (5)	0.0657 (9)
C7	0.8755 (3)	0.30226 (19)	0.9401 (5)	0.0692 (10)
H7A	0.7992	0.2778	0.9138	0.104*
H7B	0.8788	0.3337	0.8315	0.104*
H7C	0.8881	0.3350	1.0521	0.104*
C8	1.2746 (3)	0.1548 (3)	1.0879 (7)	0.0978 (14)
H8A	1.2792	0.0986	1.0897	0.147*
H8B	1.3167	0.1750	1.2123	0.147*
H8C	1.3092	0.1750	0.9923	0.147*
C9	0.8486 (3)	0.1108 (2)	0.9370 (5)	0.0632 (9)
C10	0.6297 (3)	0.1201 (2)	0.8529 (6)	0.0735 (11)
C11	0.6041 (4)	0.0676 (3)	0.6820 (8)	0.131 (2)
H11A	0.6540	0.0222	0.7116	0.197*
H11B	0.6189	0.0952	0.5753	0.197*
H11C	0.5227	0.0517	0.6488	0.197*
C12	0.6181 (4)	0.0814 (3)	1.0336 (7)	0.1063 (15)
H12A	0.6657	0.0348	1.0584	0.159*

H12B	0.5368	0.0677	1.0175	0.159*
H12C	0.6443	0.1169	1.1403	0.159*
C13	0.5522 (3)	0.1940 (3)	0.8125 (8)	0.1084 (16)
H13A	0.5697	0.2261	0.9261	0.163*
H13B	0.4703	0.1790	0.7776	0.163*
H13C	0.5680	0.2229	0.7086	0.163*
O1	1.0949 (2)	0.38906 (15)	1.0199 (4)	0.0925 (9)
O2	1.2657 (3)	0.32986 (18)	1.0728 (6)	0.1295 (13)
O3	0.74888 (17)	0.15373 (14)	0.8891 (3)	0.0713 (8)
O4	0.85058 (19)	0.03974 (15)	0.9488 (4)	0.0876 (9)
N1	1.0615 (2)	0.12471 (17)	1.0127 (4)	0.0674 (8)
H1A	1.0730	0.0748	1.0192	0.081*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.101 (3)	0.078 (3)	0.197 (6)	-0.040 (3)	0.032 (3)	-0.010 (3)
C2	0.053 (2)	0.083 (3)	0.088 (3)	-0.0142 (19)	0.0105 (18)	0.0009 (19)
C3	0.0448 (18)	0.064 (2)	0.066 (2)	-0.0079 (15)	0.0136 (14)	0.0011 (15)
C4	0.0457 (17)	0.063 (2)	0.0539 (18)	-0.0049 (14)	0.0090 (13)	-0.0018 (14)
C5	0.0379 (16)	0.070 (2)	0.064 (2)	-0.0014 (14)	0.0084 (13)	-0.0017 (16)
C6	0.0442 (18)	0.078 (2)	0.074 (2)	-0.0119 (16)	0.0149 (15)	-0.0050 (16)
C7	0.0512 (19)	0.060 (2)	0.091 (2)	0.0005 (16)	0.0119 (17)	0.0041 (17)
C8	0.043 (2)	0.099 (3)	0.146 (4)	0.005 (2)	0.019 (2)	-0.001 (3)
C9	0.0475 (19)	0.058 (2)	0.083 (2)	-0.0048 (15)	0.0169 (16)	-0.0033 (17)
C10	0.0416 (18)	0.074 (2)	0.102 (3)	-0.0111 (16)	0.0146 (17)	-0.009 (2)
C11	0.067 (3)	0.164 (5)	0.141 (4)	-0.010 (3)	-0.005 (3)	-0.062 (4)
C12	0.075 (3)	0.098 (3)	0.152 (4)	-0.020 (2)	0.041 (3)	0.009 (3)
C13	0.050 (2)	0.110 (4)	0.159 (4)	0.008 (2)	0.018 (2)	0.002 (3)
O1	0.0673 (17)	0.0635 (16)	0.145 (3)	-0.0193 (14)	0.0268 (16)	-0.0068 (15)
O2	0.0530 (18)	0.103 (2)	0.215 (4)	-0.0239 (15)	0.0100 (19)	0.007 (2)
O3	0.0359 (12)	0.0687 (15)	0.1046 (19)	-0.0050 (10)	0.0120 (11)	0.0016 (12)
O4	0.0523 (14)	0.0607 (17)	0.144 (2)	-0.0030 (11)	0.0180 (14)	-0.0099 (14)
N1	0.0442 (15)	0.0624 (16)	0.093 (2)	-0.0020 (13)	0.0149 (13)	-0.0030 (14)

Geometric parameters (Å, °)

C1—O1	1.461 (5)	C8—H8B	0.9600
C1—H1B	0.9600	C8—H8C	0.9600
C1—H1C	0.9600	C9—O4	1.215 (4)
C1—H1D	0.9600	C9—O3	1.344 (4)
C2—O2	1.188 (4)	C10—O3	1.470 (4)
C2—O1	1.334 (4)	C10—C11	1.486 (6)
C2—C3	1.454 (5)	C10—C12	1.504 (6)
C3—C6	1.385 (5)	C10—C13	1.534 (5)
C3—C4	1.429 (4)	C11—H11A	0.9600
C4—C5	1.383 (4)	C11—H11B	0.9600
C4—C7	1.486 (4)	C11—H11C	0.9600

C5—N1	1.382 (4)	C12—H12A	0.9600
C5—C9	1.448 (4)	C12—H12B	0.9600
C6—N1	1.354 (4)	C12—H12C	0.9600
C6—C8	1.496 (5)	C13—H13A	0.9600
C7—H7A	0.9600	C13—H13B	0.9600
C7—H7B	0.9600	C13—H13C	0.9600
C7—H7C	0.9600	N1—H1A	0.8600
C8—H8A	0.9600		
O1—C1—H1B	109.5	O4—C9—O3	124.0 (3)
O1—C1—H1C	109.5	O4—C9—C5	125.0 (3)
H1B—C1—H1C	109.5	O3—C9—C5	111.0 (3)
O1—C1—H1D	109.5	O3—C10—C11	110.0 (3)
H1B—C1—H1D	109.5	O3—C10—C12	109.5 (3)
H1C—C1—H1D	109.5	C11—C10—C12	114.2 (4)
O2—C2—O1	120.2 (4)	O3—C10—C13	101.7 (3)
O2—C2—C3	126.4 (4)	C11—C10—C13	111.4 (4)
O1—C2—C3	113.4 (3)	C12—C10—C13	109.3 (3)
C6—C3—C4	108.5 (3)	C10—C11—H11A	109.5
C6—C3—C2	121.7 (3)	C10—C11—H11B	109.5
C4—C3—C2	129.8 (3)	H11A—C11—H11B	109.5
C5—C4—C3	105.7 (3)	C10—C11—H11C	109.5
C5—C4—C7	126.9 (3)	H11A—C11—H11C	109.5
C3—C4—C7	127.4 (3)	H11B—C11—H11C	109.5
N1—C5—C4	108.4 (3)	C10—C12—H12A	109.5
N1—C5—C9	117.9 (3)	C10—C12—H12B	109.5
C4—C5—C9	133.7 (3)	H12A—C12—H12B	109.5
N1—C6—C3	107.3 (3)	C10—C12—H12C	109.5
N1—C6—C8	120.0 (3)	H12A—C12—H12C	109.5
C3—C6—C8	132.6 (3)	H12B—C12—H12C	109.5
C4—C7—H7A	109.5	C10—C13—H13A	109.5
C4—C7—H7B	109.5	C10—C13—H13B	109.5
H7A—C7—H7B	109.5	H13A—C13—H13B	109.5
C4—C7—H7C	109.5	C10—C13—H13C	109.5
H7A—C7—H7C	109.5	H13A—C13—H13C	109.5
H7B—C7—H7C	109.5	H13B—C13—H13C	109.5
C6—C8—H8A	109.5	C2—O1—C1	118.1 (3)
C6—C8—H8B	109.5	C9—O3—C10	123.8 (3)
H8A—C8—H8B	109.5	C6—N1—C5	110.1 (3)
C6—C8—H8C	109.5	C6—N1—H1A	124.9
H8A—C8—H8C	109.5	C5—N1—H1A	124.9
H8B—C8—H8C	109.5		
O2—C2—C3—C6	1.3 (6)	N1—C5—C9—O4	3.7 (5)
O1—C2—C3—C6	-178.8 (3)	C4—C5—C9—O4	-176.5 (4)
O2—C2—C3—C4	-177.7 (4)	N1—C5—C9—O3	-176.4 (3)
O1—C2—C3—C4	2.2 (5)	C4—C5—C9—O3	3.4 (5)
C6—C3—C4—C5	0.6 (4)	O2—C2—O1—C1	0.1 (6)

C2—C3—C4—C5	179.7 (3)	C3—C2—O1—C1	-179.8 (3)
C6—C3—C4—C7	-179.6 (3)	O4—C9—O3—C10	2.7 (5)
C2—C3—C4—C7	-0.6 (6)	C5—C9—O3—C10	-177.2 (3)
C3—C4—C5—N1	-0.2 (3)	C11—C10—O3—C9	-63.7 (5)
C7—C4—C5—N1	-179.9 (3)	C12—C10—O3—C9	62.6 (4)
C3—C4—C5—C9	180.0 (3)	C13—C10—O3—C9	178.1 (3)
C7—C4—C5—C9	0.2 (6)	C3—C6—N1—C5	0.7 (4)
C4—C3—C6—N1	-0.8 (4)	C8—C6—N1—C5	179.4 (3)
C2—C3—C6—N1	-180.0 (3)	C4—C5—N1—C6	-0.4 (4)
C4—C3—C6—C8	-179.3 (4)	C9—C5—N1—C6	179.5 (3)
C2—C3—C6—C8	1.5 (6)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1A \cdots O4 ⁱ	0.86	2.14	2.974 (4)	165

Symmetry code: (i) $-x+2, -y, -z+2$.