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1-(3,4-Dimethoxybenzoyl)-3,5-dimethyl-1*H*-pyrazole

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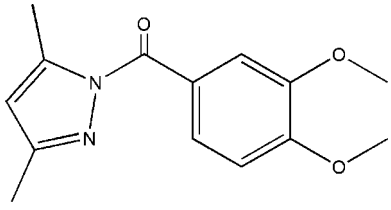
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.120; data-to-parameter ratio = 17.0.

The structure of the title compound, $C_{14}H_{16}N_2O_3$, shows the two aromatic planes twisted by an angle of 46.38 (4)°. The corresponding torsion angles are N—N—C(=O)—C of −26.7 (2)°, and C—C—C(=O)—N of −28.2 (2) and 155.5 (1) Å. The crystal packing is determined by intermolecular C—H···O hydrogen bonds, forming endless chains along [001].

Related literature

For related literature, see: Ali *et al.* (2007); Mann *et al.* (1992); Perevalov *et al.* (2001); Saeed *et al.* (2007); Smith *et al.* (1965); Soliman & Darwishl (1983); Udupi *et al.* (1998).



Experimental

Crystal data

$C_{14}H_{16}N_2O_3$
M_r = 260.29
Triclinic, $P\overline{1}$
a = 7.703 (2) Å
b = 8.409 (2) Å
c = 11.454 (3) Å
α = 69.970 (5)°
β = 85.267 (5)°
γ = 66.655 (5)°
V = 638.8 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 120 (2) K
0.44 × 0.36 × 0.25 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.959, T_{max} = 0.976
5678 measured reflections
3011 independent reflections
2301 reflections with $I > 2\sigma(I)$
R_{int} = 0.053

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.120
S = 0.97
3011 reflections
177 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8A···N2	0.95	2.46	2.8948 (18)	108
C3—H3A···O2^{i}	0.95	2.45	3.3496 (18)	157

Symmetry code: (i) $x, y, z-1$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2002); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2191).

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supplementary materials

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1-(3,4-Dimethoxybenzoyl)-3,5-dimethyl-1*H*-pyrazole

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Comment

Pyrazoles represent an important class of five-membered nitrogen heterocycles exhibiting a wide range of biological activities (Mann *et al.*, 1992, Perevalov *et al.*, 2001). Several 1-substitued 3,5-dimethylpyrazole possess potent *in vivo* hypoglycemic activity (Soliman & Darwishl, 1983). The pyrazole ring is a major pharmacophoric substructure in a number of NSAIDs like phenylbutazone (Udupi *et al.*, 1998), oxyphenbutazone, celecoxib (Smith *et al.*, 1965) exhibiting anti-inflammatory, anti-pyretic and analgesic properties. Pyrazole derivatives being more active than the isoniazid against Mycobacterium tuberculosis have recently been reported (Ali *et al.*, 2007). Several thiourea and urea derivatives of 4-aminopyrazoles exhibit strong anticonvulsant and antituberculosis activity. In continuation of our interest in the synthesis of bioactive heterocycles (Saeed *et al.*, 2007) the synthesis of title compound was carried out by direct cyclocondensation of 3,4-dimethoxybenzyl hydrazide with 2,4-pentanedione.

Experimental

A mixture of 3,4-Dimethoxybenzyl hydrazide (1 mmol) and 2,4-pentanedione (1 mmol) was refluxed in dry ethanol for 5 h. On completion of the reaction, followed by TLC examination using hexane ethyl acetate (8:2) the solvent was evaporated and reaction mixture diluted with ethyl acetate and subjected directly to thick layer chromatography on silica gel. Recrystallization from ethyl acetate yielded colourless crystals, m.p.105–107°C. IR (KBr) cm^{-1}: 3103, 2958, 2930, 1680, 1601, 1584, 1472, 1373, 1275, 1170, 1022; ^{1}H NMR ($CDCl_3$) δ 7.2 (d, 8.4, 1H), 6.89 (dd, 1H), 6.7 (dd, 8.2, 3.4, 1H). 6.02 (s, 1H), 2.83 (s, 3H), 2.26 (s, 3H); EIMS m/e 260 [M^+], 165 (base).

Refinement

Hydrogen atoms were located in difference syntheses, refined at idealized positions riding on the C (C–H = 0.95–0.99 Å) atoms with isotropic displacement parameters U_{iso}(H) = 1.2U(C_{eq}) and 1.5(methyl-C). Methyl H atoms were refined on the basis of rigid groups allowed to rotate but not tip.

Figures

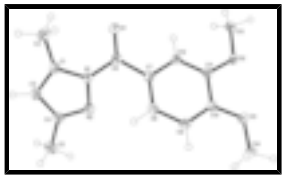

Fig. 1. Molecular structure of I. Displacement ellipsoids are drawn at the 50% probability level.

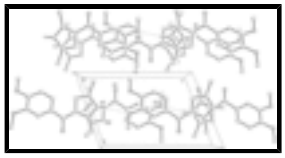

Fig. 2. Crystal packing of I viewed along [100] with hydrogen bond indicated as dashed lines. H-atoms not involved are omitted.

1-(3,4-Dimethoxybenzoyl)-3,5-dimethyl-1*H*-pyrazole

Crystal data

$C_{14}H_{16}N_2O_3$	$Z = 2$
$M_r = 260.29$	$F_{000} = 276$
Triclinic, $P\overline{1}$	$D_x = 1.353$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.703$ (2) Å	Cell parameters from 719 reflections
$b = 8.409$ (2) Å	$\theta = 2.8$–28.3°
$c = 11.454$ (3) Å	$\mu = 0.10$ mm^{-1}
$\alpha = 69.970$ (5)°	$T = 120$ (2) K
$\beta = 85.267$ (5)°	Prism, colourless
$\gamma = 66.655$ (5)°	0.44 × 0.36 × 0.25 mm
$V = 638.8$ (3) Å^3	

Data collection

Bruker SMART APEX diffractometer	3011 independent reflections
Radiation source: sealed tube	2301 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.053$
$T = 120$(2) K	$\theta_{max} = 27.9^\circ$
φ and ω scans	$\theta_{min} = 1.9^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2002)	$h = -8 \rightarrow 10$
$T_{min} = 0.959$, $T_{max} = 0.976$	$k = -10 \rightarrow 11$
5678 measured reflections	$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	Hydrogen site location: difference Fourier map
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.045$	$w = 1/[\sigma^2(F_o^2) + (0.0704P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.120$	$(\Delta/\sigma)_{max} < 0.001$
$S = 0.97$	$\Delta\rho_{max} = 0.28$ e Å^{-3}
3011 reflections	$\Delta\rho_{min} = -0.28$ e Å^{-3}
177 parameters	Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Extinction coefficient: 0.013 (2)

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}
O1	0.35654 (16)	0.20752 (14)	0.29144 (9)	0.0318 (3)
O2	0.20118 (14)	0.44061 (12)	0.66198 (8)	0.0244 (2)
O3	0.23018 (14)	0.75359 (13)	0.58447 (9)	0.0256 (2)
N1	0.26483 (17)	0.46669 (15)	0.12170 (10)	0.0232 (3)
N2	0.13919 (17)	0.64924 (15)	0.07561 (10)	0.0253 (3)
C1	0.3239 (2)	0.3922 (2)	0.02736 (12)	0.0256 (3)
C2	0.4637 (2)	0.2008 (2)	0.04724 (15)	0.0346 (4)
H2A	0.4951	0.1837	−0.0335	0.052*
H2B	0.4090	0.1134	0.0969	0.052*
H2C	0.5788	0.1794	0.0914	0.052*
C3	0.2365 (2)	0.5296 (2)	−0.07940 (13)	0.0298 (3)
H3A	0.2491	0.5231	−0.1610	0.036*
C4	0.1224 (2)	0.6848 (2)	−0.04548 (12)	0.0266 (3)
C5	−0.0039 (2)	0.8694 (2)	−0.12837 (14)	0.0369 (4)
H5A	−0.0778	0.9436	−0.0778	0.055*
H5B	−0.0900	0.8560	−0.1793	0.055*
H5C	0.0727	0.9305	−0.1828	0.055*
C6	0.30491 (19)	0.37422 (19)	0.25100 (12)	0.0235 (3)
C7	0.28444 (19)	0.48519 (18)	0.33182 (12)	0.0212 (3)
C8	0.30714 (19)	0.65048 (18)	0.29209 (12)	0.0231 (3)
H8A	0.3328	0.7009	0.2080	0.028*
C9	0.29251 (19)	0.74323 (18)	0.37492 (12)	0.0224 (3)
H9A	0.3094	0.8560	0.3473	0.027*
C10	0.25345 (18)	0.67149 (18)	0.49716 (12)	0.0203 (3)
C11	0.23435 (18)	0.50109 (18)	0.53931 (11)	0.0196 (3)
C12	0.25064 (18)	0.40966 (18)	0.45652 (12)	0.0202 (3)
H12A	0.2388	0.2944	0.4845	0.024*
C13	0.2007 (2)	0.26019 (18)	0.71111 (12)	0.0257 (3)
H13A	0.1006	0.2550	0.6668	0.039*
H13B	0.1775	0.2312	0.7999	0.039*

H13C	0.3238	0.1704	0.7004	0.039*
C14	0.2499 (2)	0.92627 (19)	0.54465 (14)	0.0310 (3)
H14A	0.3768	0.9098	0.5150	0.046*
H14B	0.2318	0.9719	0.6147	0.046*
H14C	0.1546	1.0153	0.4770	0.046*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0463 (7)	0.0205 (5)	0.0244 (5)	−0.0079 (5)	0.0000 (5)	−0.0083 (4)
O2	0.0340 (6)	0.0219 (5)	0.0192 (4)	−0.0116 (4)	0.0047 (4)	−0.0092 (4)
O3	0.0336 (6)	0.0208 (5)	0.0259 (5)	−0.0104 (4)	0.0020 (4)	−0.0124 (4)
N1	0.0281 (6)	0.0206 (6)	0.0195 (5)	−0.0067 (5)	0.0024 (5)	−0.0089 (5)
N2	0.0275 (6)	0.0210 (6)	0.0224 (6)	−0.0051 (5)	−0.0014 (5)	−0.0059 (5)
C1	0.0318 (8)	0.0292 (8)	0.0238 (7)	−0.0159 (6)	0.0067 (6)	−0.0147 (6)
C2	0.0410 (9)	0.0303 (8)	0.0354 (8)	−0.0124 (7)	0.0123 (7)	−0.0188 (7)
C3	0.0404 (9)	0.0343 (8)	0.0219 (7)	−0.0199 (7)	0.0044 (6)	−0.0127 (6)
C4	0.0308 (8)	0.0277 (8)	0.0228 (7)	−0.0143 (6)	−0.0008 (6)	−0.0065 (6)
C5	0.0416 (9)	0.0335 (9)	0.0284 (8)	−0.0130 (7)	−0.0082 (7)	−0.0018 (7)
C6	0.0251 (7)	0.0228 (7)	0.0209 (6)	−0.0069 (6)	0.0019 (5)	−0.0084 (6)
C7	0.0205 (6)	0.0205 (7)	0.0203 (6)	−0.0041 (5)	−0.0013 (5)	−0.0082 (5)
C8	0.0229 (7)	0.0244 (7)	0.0182 (6)	−0.0074 (6)	0.0005 (5)	−0.0049 (5)
C9	0.0222 (7)	0.0181 (6)	0.0248 (7)	−0.0073 (5)	−0.0012 (5)	−0.0050 (5)
C10	0.0183 (6)	0.0187 (6)	0.0230 (6)	−0.0039 (5)	−0.0031 (5)	−0.0091 (5)
C11	0.0177 (6)	0.0191 (6)	0.0183 (6)	−0.0036 (5)	−0.0006 (5)	−0.0056 (5)
C12	0.0203 (6)	0.0166 (6)	0.0219 (6)	−0.0047 (5)	−0.0006 (5)	−0.0068 (5)
C13	0.0337 (8)	0.0201 (7)	0.0211 (6)	−0.0099 (6)	0.0047 (6)	−0.0059 (5)
C14	0.0365 (8)	0.0219 (7)	0.0386 (8)	−0.0114 (6)	0.0007 (7)	−0.0148 (6)

Geometric parameters (Å, °)

O1—C6	1.2171 (17)	C5—H5B	0.9800
O2—C11	1.3610 (15)	C5—H5C	0.9800
O2—C13	1.4274 (16)	C6—C7	1.4844 (18)
O3—C10	1.3616 (15)	C7—C8	1.3841 (19)
O3—C14	1.4314 (16)	C7—C12	1.3998 (18)
N1—N2	1.3869 (16)	C8—C9	1.3931 (18)
N1—C1	1.3870 (16)	C8—H8A	0.9500
N1—C6	1.4106 (17)	C9—C10	1.3823 (18)
N2—C4	1.3209 (17)	C9—H9A	0.9500
C1—C3	1.355 (2)	C10—C11	1.4105 (18)
C1—C2	1.489 (2)	C11—C12	1.3818 (17)
C2—H2A	0.9800	C12—H12A	0.9500
C2—H2B	0.9800	C13—H13A	0.9800
C2—H2C	0.9800	C13—H13B	0.9800
C3—C4	1.418 (2)	C13—H13C	0.9800
C3—H3A	0.9500	C14—H14A	0.9800
C4—C5	1.484 (2)	C14—H14B	0.9800
C5—H5A	0.9800	C14—H14C	0.9800

C11—O2—C13	116.84 (9)	C8—C7—C12	119.57 (12)
C10—O3—C14	116.39 (10)	C8—C7—C6	123.72 (12)
N2—N1—C1	111.43 (11)	C12—C7—C6	116.61 (12)
N2—N1—C6	120.77 (10)	C7—C8—C9	120.30 (12)
C1—N1—C6	127.52 (12)	C7—C8—H8A	119.9
C4—N2—N1	104.43 (11)	C9—C8—H8A	119.9
C3—C1—N1	105.93 (13)	C10—C9—C8	120.15 (12)
C3—C1—C2	130.03 (13)	C10—C9—H9A	119.9
N1—C1—C2	123.97 (13)	C8—C9—H9A	119.9
C1—C2—H2A	109.5	O3—C10—C9	124.91 (12)
C1—C2—H2B	109.5	O3—C10—C11	115.09 (11)
H2A—C2—H2B	109.5	C9—C10—C11	120.00 (11)
C1—C2—H2C	109.5	O2—C11—C12	125.47 (12)
H2A—C2—H2C	109.5	O2—C11—C10	115.23 (11)
H2B—C2—H2C	109.5	C12—C11—C10	119.30 (11)
C1—C3—C4	106.59 (12)	C11—C12—C7	120.64 (12)
C1—C3—H3A	126.7	C11—C12—H12A	119.7
C4—C3—H3A	126.7	C7—C12—H12A	119.7
N2—C4—C3	111.61 (13)	O2—C13—H13A	109.5
N2—C4—C5	120.63 (13)	O2—C13—H13B	109.5
C3—C4—C5	127.76 (13)	H13A—C13—H13B	109.5
C4—C5—H5A	109.5	O2—C13—H13C	109.5
C4—C5—H5B	109.5	H13A—C13—H13C	109.5
H5A—C5—H5B	109.5	H13B—C13—H13C	109.5
C4—C5—H5C	109.5	O3—C14—H14A	109.5
H5A—C5—H5C	109.5	O3—C14—H14B	109.5
H5B—C5—H5C	109.5	H14A—C14—H14B	109.5
O1—C6—N1	118.94 (12)	O3—C14—H14C	109.5
O1—C6—C7	122.82 (12)	H14A—C14—H14C	109.5
N1—C6—C7	118.23 (12)	H14B—C14—H14C	109.5
C1—N1—N2—C4	0.04 (15)	N1—C6—C7—C12	155.47 (12)
C6—N1—N2—C4	−174.31 (12)	C12—C7—C8—C9	−1.3 (2)
N2—N1—C1—C3	0.66 (15)	C6—C7—C8—C9	−177.61 (12)
C6—N1—C1—C3	174.54 (12)	C7—C8—C9—C10	−0.6 (2)
N2—N1—C1—C2	177.94 (12)	C14—O3—C10—C9	−0.54 (19)
C6—N1—C1—C2	−8.2 (2)	C14—O3—C10—C11	179.64 (11)
N1—C1—C3—C4	−1.04 (16)	C8—C9—C10—O3	−177.77 (12)
C2—C1—C3—C4	−178.10 (14)	C8—C9—C10—C11	2.0 (2)
N1—N2—C4—C3	−0.72 (15)	C13—O2—C11—C12	6.10 (19)
N1—N2—C4—C5	179.43 (12)	C13—O2—C11—C10	−173.65 (11)
C1—C3—C4—N2	1.15 (17)	O3—C10—C11—O2	−1.92 (17)
C1—C3—C4—C5	−179.01 (14)	C9—C10—C11—O2	178.25 (12)
N2—N1—C6—O1	154.03 (13)	O3—C10—C11—C12	178.31 (11)
C1—N1—C6—O1	−19.3 (2)	C9—C10—C11—C12	−1.52 (19)
N2—N1—C6—C7	−26.76 (18)	O2—C11—C12—C7	179.82 (12)
C1—N1—C6—C7	159.88 (13)	C10—C11—C12—C7	−0.4 (2)
O1—C6—C7—C8	151.01 (14)	C8—C7—C12—C11	1.9 (2)
N1—C6—C7—C8	−28.17 (19)	C6—C7—C12—C11	178.39 (12)

O1—C6—C7—C12	−25.4 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8A···N2	0.95	2.46	2.8948 (18)	108
C3—H3A···O2^{i}	0.95	2.45	3.3496 (18)	157

Symmetry codes: (i) *x*, *y*, *z*−1.

Fig. 1

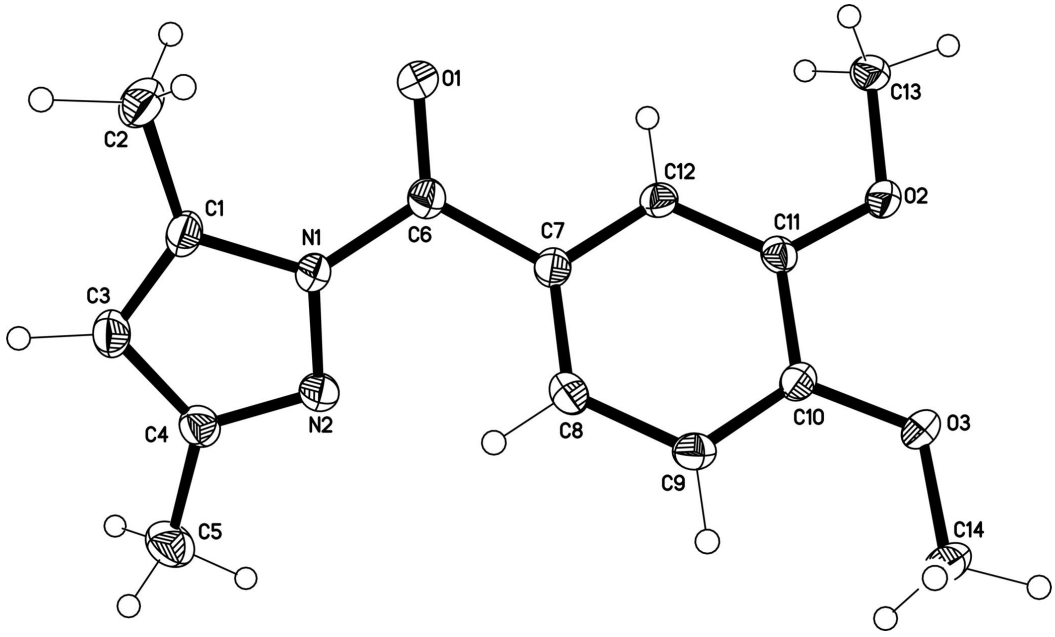

Fig. 2