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Dimethyl 3-(*tert*-butylamino)-5-oxo-7-phenyl-1*H*,5*H*-pyrazolo[1,2-*a*]pyrazole-1,2-dicarboxylate

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Key indicators

Single-crystal X-ray study
T = 290 K
Mean σ(C–C) = 0.002 Å
R factor = 0.037
wR factor = 0.105
Data-to-parameter ratio = 16.9

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

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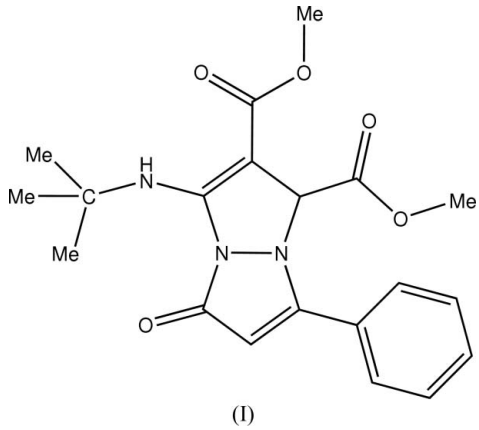
In the title compound, $C_{20}H_{23}N_3O_5$, the molecule contains two pyrazole rings with an interplanar angle of 34.1 (1)°. The phenyl and pyrazolone rings make a dihedral angle of 35.43 (13)° with one another. There is an intramolecular hydrogen bond between the carbonyl O atom in one of the carboxylate groups and the N atom of the *tert*-butylamine substituent of the pyrazole ring. This is the first structure reported of a polyfunctional pyrazolo[1,2-*a*]pyrazole derivative of potential synthetic and pharmaceutical interest.

Comment

The structure of the title compound has been studied because pyrazolo[1,2-*a*]pyrazoles have been shown to possess useful biological activities (Radl, 1996). We have recently published a facile synthesis of highly functionalized pyrazolo[1,2-*a*]pyrazoles *via* a one-pot isocyanide-based three-component reaction (Adib *et al.*, 2005), and we now report the structure of the title compound, (I).



(I)

The molecular structure of (I) and the atom-numbering scheme are shown in Fig. 1. The phenyl and pyrazolone rings make a dihedral angle of 35.43 (13)° with one another. An intramolecular hydrogen bond is formed between the carbonyl O atom of the 2-carboxylate (O2) and the N atom of the *tert*-butylamine (N1), giving rise to a slightly puckered six-membered chelate ring (Table 2). A noticeable degree of conjugation in this chelate ring makes the 2-carboxylate coplanar with the pyrazole ring.

Experimental

Compound (I) was synthesized according to the literature (Adib *et al.*, 2005). Crystals suitable for crystallographic analysis were obtained (yield 0.27 g, 70%) after recrystallization from 1:1 hexane–ethyl acetate.

Crystal data

$C_{20}H_{23}N_3O_5$
M_r = 385.41
Monoclinic, $P2_1/n$
a = 12.1826 (8) Å
b = 9.4017 (6) Å
c = 17.1789 (12) Å
β = 91.059 (6)°
V = 1967.3 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 290 (2) K
0.5 × 0.2 × 0.2 mm

Data collection

Oxford Xcalibur3 CCD diffractometer
Absorption correction: numerical (*X-RED*; Stoe & Cie, 1997)
T_{min} = 0.976, T_{max} = 0.995
13972 measured reflections
4282 independent reflections
2751 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.105
S = 1.01
4282 reflections
254 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Selected torsion angles (°).

C14—N2—N3—C11	152.35 (10)	C14—N2—N3—C12	2.23 (13)
C9—N2—N3—C11	14.73 (12)	N2—C14—C15—C20	144.47 (13)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2	0.86	2.24	2.7910 (14)	122
C6—H6*C*···O5	0.96	2.37	3.193 (2)	143
C6—H6*C*···O5	0.96	2.37	3.193 (2)	143
C7—H7*A*···O2^{i}	0.96	2.59	3.5461 (19)	175
C8—H8*B*···O5	0.96	2.41	3.225 (2)	142
C16—H16···O2^{ii}	0.93	2.51	3.4422 (18)	178

Symmetry codes: (i) $-x+1, -y, -z$; (ii) $-x+1, -y+1, -z$.

All H atoms were positioned at calculated positions and constrained to ride on their parent atoms, with C—H = 0.93–0.98 Å, N—H = 0.86 Å, U_{iso}(H) = 1.2U_{eq}(C,N) (for CH and NH groups) and U_{iso}(H) = 1.5U_{eq}(C) (for CH_3).

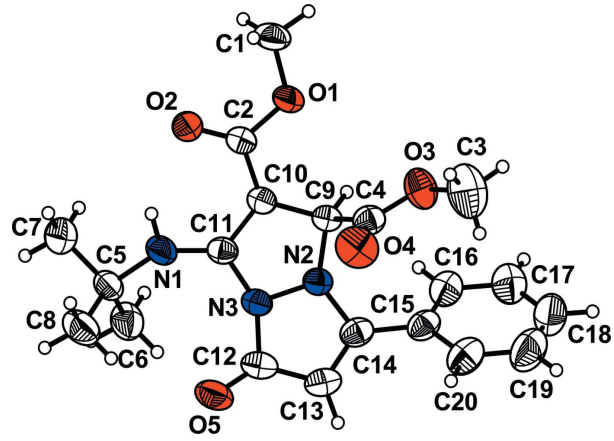


Figure 1
Molecular structure of (I), showing the atom numbering scheme, with 50% probability displacement ellipsoids. H atoms are shown as circles of arbitrary radius.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2003); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXL97*.

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