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Key indicators

Single-crystal X-ray study
 $T = 93\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.051
 wR factor = 0.141
Data-to-parameter ratio = 12.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.***tert*-Butyl 1,4-dioxo-3,6-diphenyl-1,2,4,5-tetra-
hydropyrrolo[3,4-*c*]pyrrole-2-carboxylate**

The title compound, $\text{C}_{23}\text{H}_{20}\text{N}_2\text{O}_4$, is a soluble precursor ('latent pigment') of diketopyrrolopyrrole pigments. The *tert*-butoxycarbonyl group is twisted by $78.5(1)^\circ$ with respect to the heterocyclic ring system. In the crystal structure, pairs of molecules form centrosymmetric dimers through $\text{N}-\text{H}\cdots\text{O}$ intermolecular hydrogen bonds and are stacked in a herringbone fashion along the a axis.

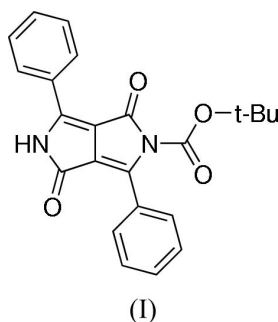
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Comment

The title compound, (I), is a soluble precursor ('latent pigment') (Zambounis *et al.*, 1994, 1997) of diketopyrrolopyrrole (DPP), an industrially important red pigment (Herbst & Hunger, 1993). The soluble precursor is prepared by replacing the H atom of the NH group by a *tert*-butoxycarbonyl (*t*-BOC) group. Both mono- and disubstituted *t*-BOC DPPs can be prepared. The former is called mono-BOC DPP while the latter is simply *t*-BOC DPP. The insoluble parent DPP can then be regenerated by thermal decomposition of the precursor at around 450 K. The present 'latent pigment technology' has recently attracted attention as a versatile and promising technique for the preparation of pigment nanoparticles as well as transparent thin films for electronic and imaging applications. The structure of *t*-BOC DPP has previously been published (MacLean *et al.*, 2000; Mizuguchi, 2003*a,b*). The present paper reports the structure of mono-BOC DPP, (I).



The title compound is shown in Fig. 1. The phenyl rings deviate asymmetrically from the heterocyclic system in the same direction by $4.4(1)^\circ$ ($\text{N1}/\text{C1}/\text{C2}/\text{C5}/\text{C6}$ and $\text{C7}/\text{C8}/\text{C9}/\text{C10}/\text{C11}/\text{C12}$) and $9.3(1)^\circ$ ($\text{N2}/\text{C1}/\text{C2}/\text{C3}/\text{C4}$ and $\text{C13}/\text{C14}/\text{C15}/\text{C16}/\text{C17}/\text{C18}$). The *t*-BOC group attached to the N atom of the heterocyclic ring is twisted with respect to the heterocyclic system by $78.5(1)^\circ$ ($\text{N2}/\text{C1}/\text{C2}/\text{C3}/\text{C4}$ and $\text{O3}/\text{O4}/\text{C19}/\text{C20}$). Furthermore, the heterocyclic ring system is not entirely planar, but is folded in the middle with a dihedral angle of $177.6(1)^\circ$ between the two five-membered rings. Fig. 2 shows

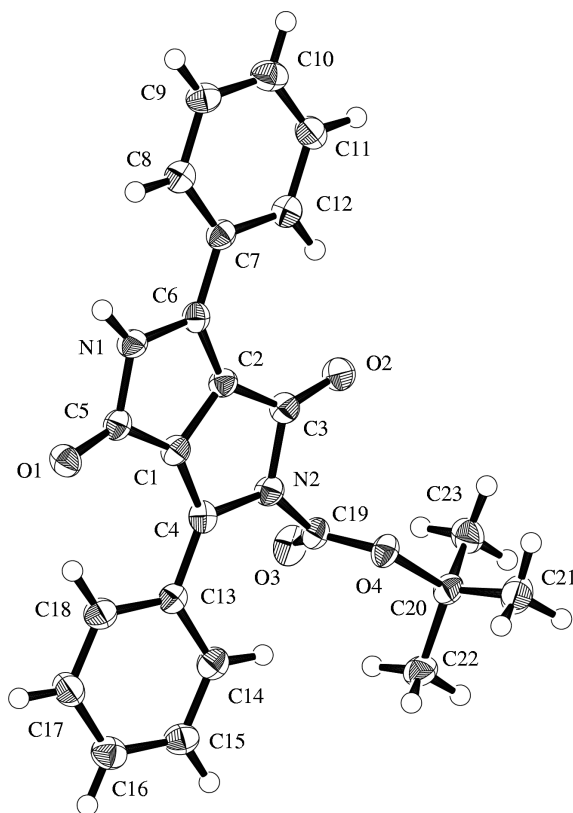


Figure 1
A view of the molecular structure of (I), showing 50% displacement ellipsoids for non-H atoms.

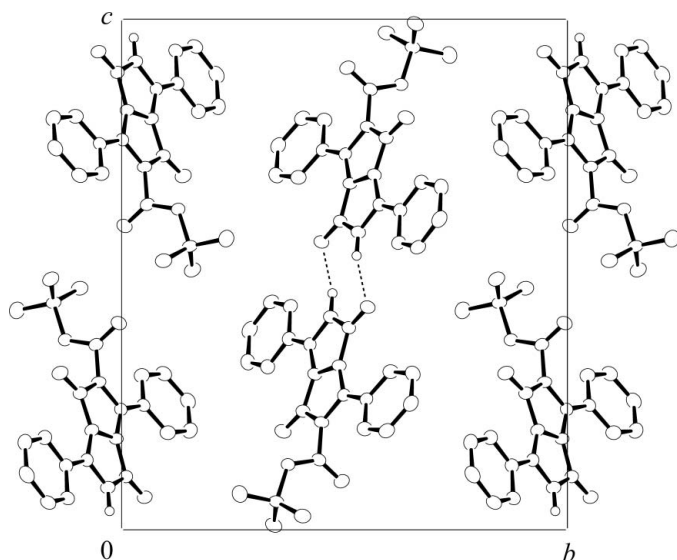


Figure 2
Projection on to the *bc* plane, showing the N—H...O intermolecular hydrogen bonds as dotted lines. H atoms not involved in hydrogen bonding have been omitted.

the projection of the structure on to the *bc* plane. In the crystal structure, pairs of molecules form centrosymmetric dimers through N—H...O intermolecular hydrogen bonds between the NH group of one molecule and the carbonyl group of a neighboring molecule. However, as shown in Fig. 3, there is a small step of about 0.84 Å between the two molecular planes

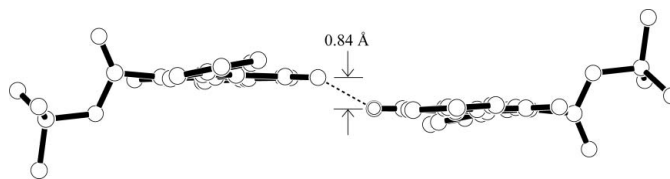


Figure 3
Side view of the hydrogen-bonded molecules, showing a step of about 0.84 Å. H atoms have been omitted.

of a centrosymmetric pair. The molecules are stacked in a herring-bone fashion along the *a* axis.

Experimental

Mono-BOC DPP was prepared according to a method described in the literature (Zambounis *et al.*, 1994). The product was purified by column chromatography. Single crystals of mono-BOC DPP were grown from a dichloromethane–ethyl acetate solution.

Crystal data

$C_{23}H_{20}N_2O_4$
 $M_r = 388.41$
Monoclinic, $P2_1/n$
 $a = 6.0502$ (6) Å
 $b = 16.524$ (2) Å
 $c = 19.031$ (2) Å
 $\beta = 95.744$ (7)°
 $V = 1893.0$ (4) Å³
 $Z = 4$

$D_x = 1.363$ Mg m⁻³
Cu $K\alpha$ radiation
Cell parameters from 11787 reflections
 $\theta = 3.5$ – 68.1 °
 $\mu = 0.77$ mm⁻¹
 $T = 93.2$ K
Needle, orange
0.50 × 0.10 × 0.05 mm

Data collection

Rigaku R-Axis RAPID imaging plate diffractometer
 ω scans
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.683$, $T_{\max} = 0.962$
17683 measured reflections

3329 independent reflections
2370 reflections with $F^2 > 2\sigma(F^2)$
 $R_{\text{int}} = 0.052$
 $\theta_{\max} = 68.3$ °
 $h = -6 \rightarrow 6$
 $k = -19 \rightarrow 19$
 $l = -22 \rightarrow 22$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.141$
 $S = 1.08$
3329 reflections
265 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + \{0.05[\text{Max}(F_o^2, 0) + 2F_c^2/3]^2\}]$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.28$ e Å⁻³
 $\Delta\rho_{\min} = -0.31$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

N1—C5	1.395 (3)	C1—C4	1.382 (3)
N1—C6	1.395 (3)	C1—C5	1.455 (3)
N2—C3	1.427 (3)	C2—C3	1.449 (3)
N2—C4	1.404 (3)	C2—C6	1.379 (3)
C1—C2	1.425 (3)		
C5—N1—C6	112.6 (2)	C1—C2—C6	109.5 (2)
C3—N2—C4	112.7 (2)	C3—C2—C6	142.2 (2)
C2—C1—C4	110.1 (2)	N2—C3—C2	103.1 (2)
C2—C1—C5	107.3 (2)	N2—C4—C1	105.7 (2)
C4—C1—C5	142.5 (2)	N1—C5—C1	104.2 (2)
C1—C2—C3	108.3 (2)	N1—C6—C2	106.4 (2)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1N\cdots O1^i$	0.94 (2)	1.90 (2)	2.821 (2)	166 (2)

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

The H atom attached to the N atom was located in a difference density map and its coordinates were refined, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$. The remainder of the H atoms were positioned geometrically and included in a riding-model approximation with $C-H = 0.95 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *TEXSAN* (Molecular Structure Corporation, 2001); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structure:

TEXSAN; molecular graphics: *ORTEP III* (Burnett & Johnson, 1996); software used to prepare material for publication: *TEXSAN*.

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