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N-(*tert*-Butyloxycarbonylamino)phthalimide

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean *σ*(C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.129
Data-to-parameter ratio = 14.1

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

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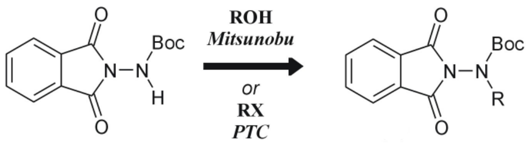
An X-ray crystallographic study reveals a short N—N bond in the title compound, $C_{13}H_{14}N_2O_4$. The molecular packing is mainly driven by strong N—H$\cdots$O═C hydrogen bonds, leading to *C*(5) infinite chains, and by π–π stacking interactions between the phthalimide ring systems.

Comment

As part of our effort to develop new synthetic approaches for the preparation of hydrazine derivatives, we had previously demonstrated that *N*-*tert*-butyloxycarbonylaminophthalimide, (I) [easily prepared from *tert*-butylcarbazate and phthalic anhydride (Brosse *et al.*, 2000)], can be used for the synthesis of protected alkylhydrazines (II). As a result, we showed that (I) can be considered as a hydrazine bearing three electron-withdrawing groups, two of which are incorporated into the phthaloyl moiety. This structural arrangement enabled us to use this hydrazine derivative as an acidic partner in phase transfer catalysis (PTC) procedures (Brosse *et al.*, 2003) and, for the first time, in the Mitsunobu protocol (Brosse *et al.*, 2000). This alkylation method was efficient in obtaining, in two steps, *N*-alkylated hydrazines and enantiomerically pure α-hydrazinoesters (Brosse *et al.*, 2001).



(I) (II)

Compound (I) crystallizes in the centrosymmetric monoclinic space group $P2_1/c$. Bond lengths and angles of both phthalimide and urethane groups are consistent with those reported for similar groups (Allen, 2002). The angle between the mean planes defined by the urethane and phthalimide groups is 82.70 (6)°, showing that these two groups are nearly perpendicular. A search of the Cambridge Structural Database (CSD; Version 5.25; Allen, 2002) for crystal structures containing the *N*-aminophthalimide group, yielded only 36

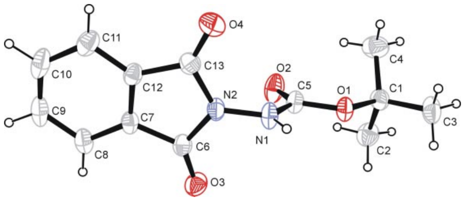


Figure 1
An *ORTEP*-3 (Farrugia, 1997) drawing of (I), showing the atom-numbering scheme and 25% probability displacement ellipsoids.

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hits, and revealed that N—N bond lengths range from 1.37 to 1.43 Å. The value observed in (I), 1.372 (2) Å, is in agreement with the results of Loehlin (1985), which assumed that the N—N bond length is dependent on lone-pair localization. Indeed, the partial electron delocalization from both atoms N1 and N2 results in a short distance.

In the crystal structure, molecules of (I) are linked into infinite chains parallel to *b* *via* C═O···H—N hydrogen bonds (Fig. 2 and Table 1) described by the pattern *C*(5) (Bernstein *et al.*, 1995). The phthalimide rings interact in pairs *via* π–π stacking interactions, with a distance between the two ring centroids of 3.637 (1) Å. All other intermolecular interactions are van der Waals interactions.

Experimental

The title compound was prepared from *tert*-butylcarbazate and phthalic anhydride (Brosse *et al.*, 2000), and was crystallized by slow evaporation of an ethanol solution.

Crystal data

$C_{13}H_{14}N_2O_4$
$M_r = 262.26$
Monoclinic, $P2_1/c$
$a = 9.7050\ (6)$ Å
$b = 8.5290\ (4)$ Å
$c = 16.3120\ (10)$ Å
$\beta = 95.275\ (3)^\circ$
$V = 1344.49\ (13)$ Å^3
$Z = 4$
$D_x = 1.296$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 10 577 reflections
$\theta = 3.4$–25.4°
$\mu = 0.10$ mm^{-1}
$T = 293\ (2)$ K
Prism, colourless
0.2 × 0.1 × 0.1 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: none
11 015 measured reflections
2444 independent reflections
1701 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.033$
$\theta_{\max} = 25.4^\circ$
$h = -11 \rightarrow 11$
$k = -10 \rightarrow 10$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.129$
$S = 1.04$
2444 reflections
173 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0577P)^2 + 0.2922P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.20$ e Å^{-3}
$\Delta\rho_{\min} = -0.13$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.044 (5)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3$^{\rm i}$	0.86	2.10	2.831 (2)	143

Symmetry code: (i) $-x, y-\frac{1}{2}, \frac{1}{2}-z$.

All H atoms were placed at calculated positions and refined using a riding model, with C—H distances of 0.93–0.96 Å and an N—H distance of 0.86 Å. The H-atom $U_{\rm iso}$ parameters were fixed at $1.2U_{\rm eq}$(C) for aromatic C—H groups, at $1.2U_{\rm eq}$(N) for the N—H group and at $1.5U_{\rm eq}$(C) for methyl C—H.

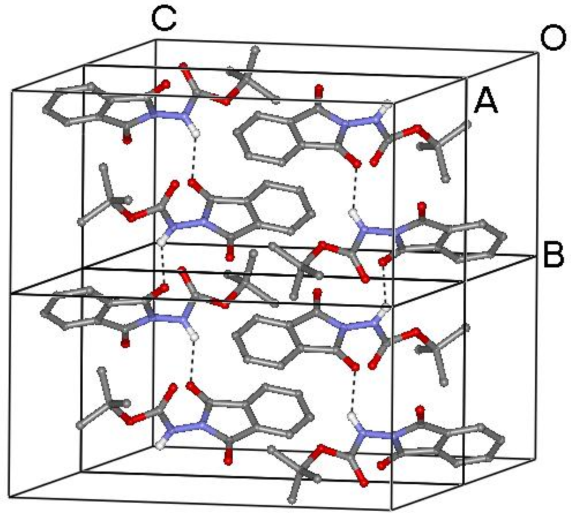

Figure 2
Part of the crystal structure of (I), showing the formation of the *C*(5) chains along [010]. The intermolecular hydrogen bonds are shown as dashed lines.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *COLLECT*; data reduction: *HKL* suite (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997) and *WebLab ViewerPro* 3.5 (MSI, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2003).

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