organic papers

# N,N,N′-Triacetyl-2,6-diamino-4-*tert*-butylanisole

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Key indicators

Single-crystal X-ray study
T = 93 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.034
wR factor = 0.089
Data-to-parameter ratio = 8.9

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

The solid state structure of the title compound, $C_{17}H_{24}N_2O_4$, reveals the imide unit to be planar and perpendicular to the plane of the benzene ring. In the crystal structure, adjacent molecules are hydrogen bonded through the amide unit.

Comment

The search for binucleating ligands which give interesting metal–metal interactions has brought us to investigate diazaanthracenols. These can be synthesized from the corresponding diaminophenols *via* the Skraup reaction (Manske & Kulka, 1953). To slow down the notoriously fast Skraup reaction, the amine groups may be acetylated. During the course of such an acetylation, we fortuitously synthesized the title compound, (I), which crystallizes with one molecule in the asymmetric unit (Fig. 1).
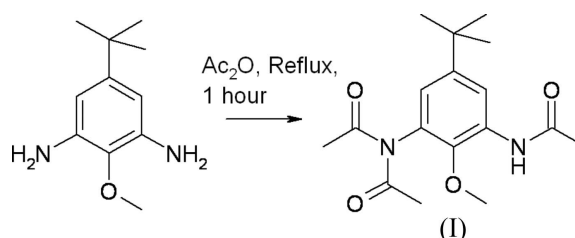
The imide unit of the molecule is essentially planar, and perpendicular to the plane of the central benzene ring [81.44 (2)°]. In contrast, the amide unit is approximately coplanar with the benzene ring [12.0 (2)°], with the NH hydrogen involved in an intramolecular N2—H2···O1 hydrogen bond to the methoxy O atom. Molecules are linked together by a long intermolecular N2—H2···O4[i] hydrogen bond between the monoacetyl units (Fig. 2 and Table 1).

Experimental

The title compound was prepared by refluxing the free base (2,6-diamino-4-*tert*-butylanisole, 1 g) in excess acetic anhydride (50 ml). After 1 h, water (50 ml) was added to the reaction mixture and the mixture was left to cool. Large blocks of (I) were found the next morning (yield 0.50 g, 30%). ^{1}H NMR (CDCl$_3$): δ 1.31 [9H, s, (CH$_3$)$_3$], 2.22 (3H, s, NHCOCH$_3$), 2.31 [6H, s, N(COCH$_3$)$_2$], 3.70 (3H, s, OCH$_3$), 6.80 (1H, s, ArH), 7.68 (1H, s, NH), 8.49 (1H, s, ArH); ^{13}C NMR (CDCl$_3$): δ 24.9 (NCOCH$_3$), 26.3 (N(COCH$_3$)$_2$), 31.2 [C(CH$_3$)$_3$], 34.8 [ArC(CH$_3$)$_3$], 61.0 (OCH$_3$), 118.8 (ArCH), 121.4 (ArCH), 127.8 (ArCOCH$_3$), 132.1 (ArCNCOCH$_3$), 148.8 [ArCC(CH$_3$)$_3$], 168.4 [ArCN(COCH$_3$)$_2$], 172.9 (NCOCH$_3$) and 173.4 [N(COCH$_3$)$_2$]. Analysis calculated for $C_{17}H_{24}N_2O_4$: C 63.73, H 7.55, N 8.74%; found: C 63.74, H 7.61, N 8.68%.

Crystal data

$C_{17}H_{24}N_2O_4$
$M_r = 320.38$
Orthorhombic, $Pna2_1$
$a = 9.9409$ (6) Å
$b = 20.9722$ (14) Å
$c = 8.4392$ (6) Å
$V = 1759.4$ (2) Å^3
$Z = 4$
$D_x = 1.210$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 10745
  reflections
$\theta = 2.6–26.4°$
$\mu = 0.09$ mm^{-1}
$T = 93$ (2) K
Block, colourless
$0.59 \times 0.52 \times 0.35$ mm

Data collection

Bruker APEX-II CCD area-
  detector diffractometer
φ and ω scans
Absorption correction: multi-scan
  (APEXII; Bruker, 2004)
  $T_{min} = 0.877$, $T_{max} = 0.970$
14772 measured reflections

1934 independent reflections
1858 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 26.4°$
$h = -12 \rightarrow 12$
$k = -26 \rightarrow 23$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.089$
$S = 1.06$
1934 reflections
218 parameters
H atoms treated by a mixture of
  independent and constrained
  refinement

$w = 1/[\sigma^2(F_o^2) + (0.0562P)^2 + 0.5007P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2$\cdots$O1	0.83 (3)	2.29 (3)	2.715 (3)	112 (3)
N2—H2$\cdots$O4^i	0.83 (3)	2.33 (3)	3.129 (3)	161 (3)

Symmetry code: (i) $x + \frac{1}{2}, -y + \frac{3}{2}, z$.

In the absence of significant anomalous scattering effects, Friedel pairs were averaged. The H atoms of the methyl group of the monoacetylated N atom were disordered over two sites with 50% occupancy in each site. The *tert*-butyl group showed some evidence of rotation in the form of elongated ellipsoids around the centre of rotation. This was insufficient to warrant modelling although the behaviour of C atoms with high displacement parameters were restrained to be approximately isotropic. The H atom on the N atom was found in a difference map and its position was refined with $U_{iso}(H) = 1.2U_{eq}(N2)$. All other H atoms were introduced in calculated positions as riding atoms, with distances of 1.00 (CH) and 0.98 Å (CH$_3$), and with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl groups and $U_{iso}(H) = 1.2U_{eq}(X)$, where X is any other bonded carrier atom.

Data collection: *APEXII* (Bruker 2004); cell refinement: *APEXII*; data reduction: *APEXII*; program(s) used to solve structure: *APEXII*; program(s) used to refine structure: *APEXII*; molecular graphics: *ORTEP-3* (Version 1.08; Farrugia, 1997) and *MERCURY* (Version 1.4; Bruno *et al.*, 2002); software used to prepare material for publication: *enCIFer* (Version 1.1; Allen *et al.*, 2004).
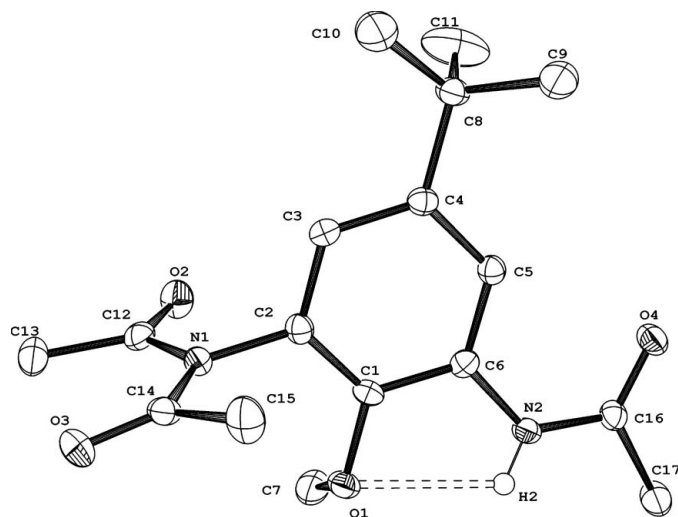
Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 50% probability level. All H atoms except for H2 have been omitted for clarity. The intramolecular hydrogen bond is shown as a double dashed line.
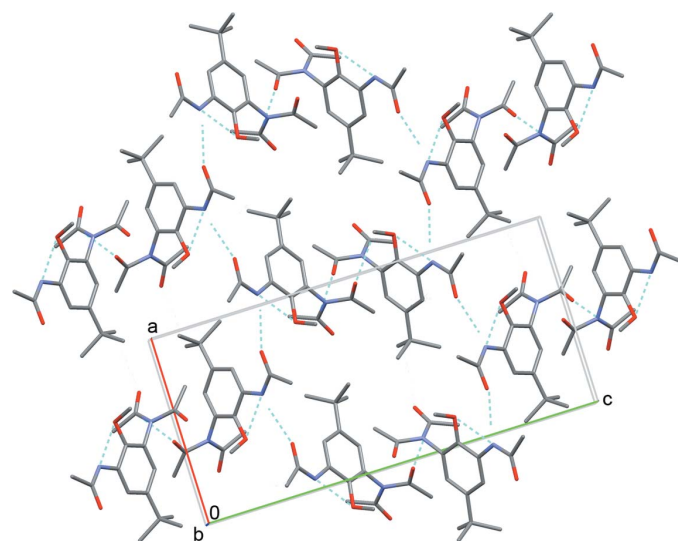

Figure 2
A packing diagram, showing the two-dimensional hydrogen-bonded sheets. H atoms have been omitted for clarity. Hydrogen bonds are drawn as dashed lines.

References

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