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2,6-Bis(triphenylacetylamino)phenol

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Key indicators

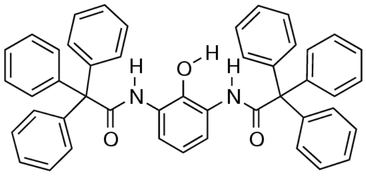
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.052
wR factor = 0.136
Data-to-parameter ratio = 17.1

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

In the title compound, $C_{46}H_{36}N_2O_3$, very bulky triphenylmethyl groups interfere with intermolecular interactions; intramolecular hydrogen bonds N—H···O—H···O═C between the phenol and two amide groups are observed in the structure.

Comment

Hydrogen-bond networks, one of the non-covalent motifs, have been employed as synthetic paradigms to rationally design crystal structures. Most interesting network structures have been reported combining intermolecular N—H···O and O—H···O hydrogen bonds, and other donor–acceptor pairs (Holman *et al.*, 2001; Noveron *et al.*, 2002; Nguyen *et al.*, 2001; Tanaka *et al.*, 2002; Mak & Xue, 2002). We have especially focused on N—H···O hydrogen bonds with various oxo acids as acceptors, such as carboxylic acids, phosphoric acids and sulfonic acids (Ueyama *et al.*, 1999; Onoda *et al.*, 2001; Onoda, Yamada, Okamura, Yamamoto & Ueyama, 2002; Onoda, Yamada, Okamura, Doi *et al.*, 2002). Thus, we have constructed a series of bulky amides with which to examine the formation of such hydrogen bonds and have determined the properties of their intramolecular hydrogen bonds. A detailed knowledge of intramolecular hydrogen bonds will provide basic information for crystal engineering. Here, we report that we have synthesized the title compound, (I), and structurally characterized the hydrogen-bond geometry.



(I)

In (I), very bulky triphenylmethyl groups interfere with intermolecular interactions; only intramolecular hydrogen bonds between the two amide groups and a phenol OH group are observed in the structure (Fig. 1 and Table 1). One amide moiety (N1—H1) is directed towards atom O3, indicating the presence of an intramolecular N—H···O hydrogen bond. The angle between the N1-amide plane and the C11—C16 aromatic ring plane is 8.67 (5)° and the other amide group participates in O—H···O═C hydrogen bonds with a phenol OH group. Actually, the N2-amide plane is twisted from the C11—C16 plane by an angle of 36.91 (5)° in order to form a seven-membered hydrogen-bonded ring.

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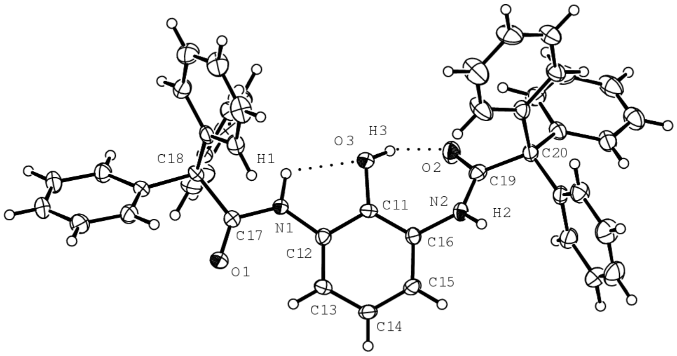

Figure 1
The molecular structure of (I), showing the labeling of selected non-H atoms and 30% probability displacement ellipsoids.

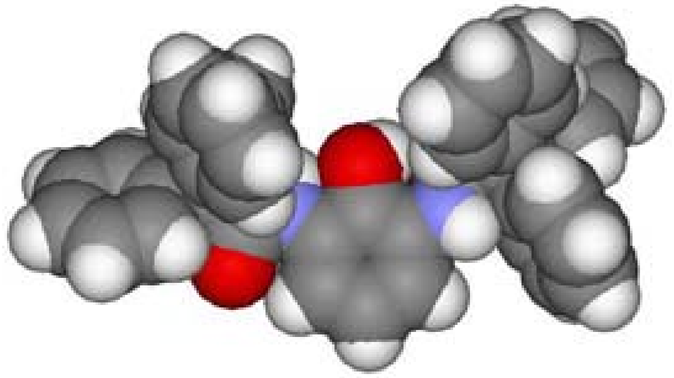

Figure 2
CPK drawing of the molecular structure of (I). Key: red (O), blue (N), black (C) and white (H).

The IR spectrum of (I) in the solid state (KBr pellet) shows that the OH band appears at 3458 cm^{-1}, shifted down from the free OH region. The NH bands are observed at around 3390 cm^{-1} as an overlapped signal. Thus, the intramolecular N—H···O hydrogen bond to the phenolic OH is very weak. The ^{1}H NMR measurement in $CDCl_3$ solution (10 m*M*) shows that the amide NH signal appears at 9.11 p.p.m. at 303 K. Thus, the asymmetric orientation of the two amide groups undergo fast exchange in solution.

Experimental

The synthesis of (I) was reported previously (Onoda, Yamada, Okamura, Doi *et al.*, 2002). Crystals were obtained from a hot acetonitrile solution by slow cooling.

Crystal data

$C_{46}H_{36}N_2O_3$
$M_r = 664.77$
Monoclinic, $P2_1/c$
$a = 13.910$ (4) Å
$b = 13.425$ (4) Å
$c = 19.074$ (6) Å
$\beta = 99.074$ (19)°
$V = 3517.3$ (18) Å^3
$Z = 4$
$D_x = 1.255$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 19635 reflections
$\theta = 3.7$–55°
$\mu = 0.08$ mm^{-1}
$T = 296$ K
Prism, colorless
0.10 × 0.10 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID Imaging Plate diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.383$, $T_{max} = 0.992$
34 653 measured reflections
7998 independent reflections
3722 reflections with $I > 2\sigma(I)$
$R_{int} = 0.075$
$\theta_{max} = 27.5°$
$h = -18 \rightarrow 16$
$k = -17 \rightarrow 17$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.136$
$S = 0.86$
7998 reflections
469 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0697P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3	0.87 (2)	2.16 (2)	2.607 (2)	112 (2)
O3—H3···O2	0.86 (2)	1.78 (2)	2.604 (2)	159 (2)

The positional parameters of H atoms bonded to N and O atoms were refined. The N—H and O—H bond lengths are 0.86 (2)–0.87 (2) and 0.86 (2) Å, respectively. The H atoms bonded to C atoms were positioned geometrically and were treated as riding on their parent atoms, with C—H bond distances of 0.93 Å.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1991); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *TEXSAN* and *MERCURY* (Bruno *et al.*, 2002); software used to prepare material for publication: *TEXSAN* and *MERCURY*.

Support of this work by a JSPS Fellowship [for AO; grant 2306(1999–2002)] and a Grant-in-Aid for Scientific Research on Priority Area (A) (No. 10146231) from the Ministry of Education, Science, Sports and Culture, Japan, is gratefully acknowledged.

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