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4-Acetyl-*N*,*N*-dibenzoylphenylamine

Anwar Usman,[a] Ibrahim Abdul Razak,[a] Hoong-Kun Fun,[a]* Suchada Chantrapromma,[a]† Jing-Zhi Tian,[b] Yan Zhang[b] and Jian-Hua Xu[b]

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [b]Department of Chemistry, Nanjing University, Nanjing 210093, People's Republic of China

† Permanent address: Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand

Correspondence e-mail: hkfun@usm.my

Key indicators

Single-crystal X-ray study
T = 183 K
Mean σ(C–C) = 0.002 Å
R factor = 0.054
wR factor = 0.135
Data-to-parameter ratio = 14.3

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

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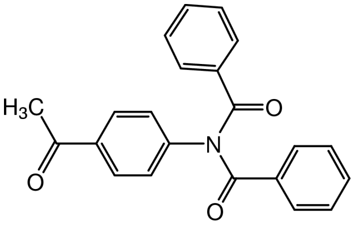
In the title compound, $C_{22}H_{17}NO_3$, the three aromatic rings are orthogonal. The crystal structure is stabilized by weak intermolecular C—H···O and C—H···π interactions.

Comment

In recent years, photo-induced electron transfer (PET) reactions of organic compounds have been extensively investigated (Scheinbaum, 1964; Kavarnos & Turro, 1986; D'Auria *et al.*, 1996). Such PET reactions of nitroaromatic compounds, which are electron acceptors, with a variety of donors are also of interest. We have recently investigated the photo-induced reactions between nitroaromatic compounds and diphenylacetylenes, which give the corresponding benzenamines as one of the products. The crystal structure of *N*,*N*-dibenzoyl-4-chloroaniline, which resulted from such a reaction between 4-chloronitrobenzene and an excess amount of 1,2-diphenylacetylene, has been reported (Usman *et al.*, 2002). We carried out a similar reaction for 4-carbomethoxynitrobenzene and isolated the title compound, (I), which was structurally analysed in order to elucidate its conformation.



(I)

The bond lengths and angles in (I) have normal values (Allen *et al.*, 1987), and are comparable with those of the related compound (Usman *et al.*, 2002). Also similar is the orthogonal disposition of the three aromatic rings, with dihedral angles between C1–C6 and C8–C13, C1–C6 and C15–C20, and C8–C13 and C15–C20 of 76.8 (1), 78.8 (1), and 80.7 (1)°, respectively. The benzoyl moieties C7–C13/O1 and C14–C20/O2 attached at atom N1 have the planes of their amide groups, O1/C7/C8/N1 and O2/C14/C15/N1, twisted from the aromatic planes by angles of 33.2 (1) and 25.6 (1)°, respectively.

The angles subtended at atom N1 (sp^3 state) joining the 4-acetylphenyl and benzoyl moieties are in the range 116.7 (1)–121.0 (1)°. The C21/C22/O3 acetyl group is twisted around the C3—C21 bond by 14.2 (1)° with respect to the attached aromatic ring.

In the crystal packing, there are two weak C—H···π interactions involving the centroids of the aromatic rings

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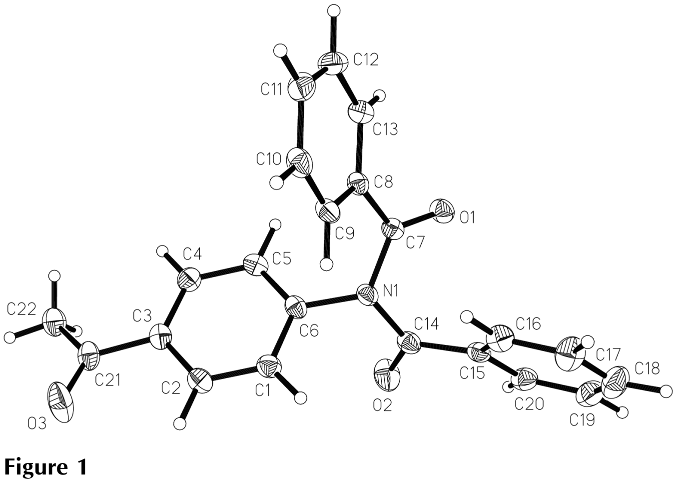

Figure 1
The structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

C8–C13 [C*g*(*B*)] and C15–C20 [C*g*(*C*)] (Table 2). The molecules are also joined by weak intermolecular C—H···O contacts (Table 2). The C22—H22*B*···O1^{iii} interaction interconnects adjacent molecules into dimers which are linked into a three-dimensional molecular network by C13—H13···O3^{ii} and C9—H9···O1^{i} interactions (Fig. 2; see Table 2 for symmetry codes).

Experimental

The title compound was prepared by the photo-induced reaction of an acetonitrile solution of 4-carbomethoxynitrobenzene, with an excess of 1,2-diphenylacetylene. The compound is one of the products of this photo-induced reaction and was isolated by column chromatography on silica gel. Single crystals were grown by slow evaporation from petroleum ether–ethyl acetate.

Crystal data

$C_{22}H_{17}NO_3$
$M_r = 343.37$
Monoclinic, $P2_1/n$
$a = 10.5190$ (1) Å
$b = 9.1443$ (2) Å
$c = 18.1947$ (3) Å
$\beta = 94.148$ (1)°
$V = 1745.54$ (5) Å^3
$Z = 4$

$D_x = 1.307$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 7550 reflections
$\theta = 3.0$–28.3°
$\mu = 0.09$ mm^{-1}
$T = 183$ (2) K
Block, colorless
0.46 × 0.30 × 0.18 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.961$, $T_{max} = 0.985$
10245 measured reflections

4192 independent reflections
3124 reflections with $I > 2\sigma(I)$
$R_{int} = 0.074$
$\theta_{max} = 28.3°$
$h = -11 \rightarrow 13$
$k = -12 \rightarrow 11$
$l = -22 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.135$
$S = 0.96$
4192 reflections
293 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0621P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.49$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.037 (3)

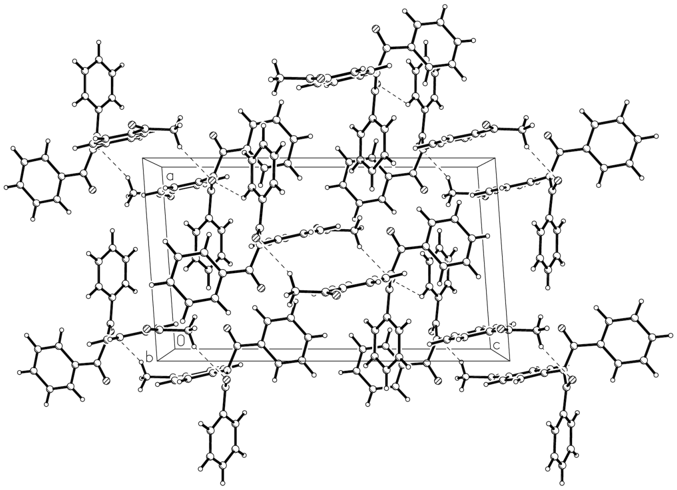

Figure 2
Packing diagram of the title compound, viewed down the *b* axis, showing the intermolecular contacts linking molecules in a three-dimensional network.

Table 1
Selected geometric parameters (Å).

O1—C7	1.216 (2)	N1—C6	1.443 (2)
O2—C14	1.208 (2)	C7—C8	1.485 (2)
N1—C7	1.411 (2)	C14—C15	1.485 (2)
N1—C14	1.426 (2)		

Table 2
Intermolecular contacts (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9···O1^{i}	1.00 (2)	2.47 (2)	3.130 (2)	123 (1)
C13—H13···O3^{ii}	1.00 (2)	2.49 (2)	3.380 (2)	148 (1)
C22—H22*B*···O1^{iii}	0.96	2.46	3.335 (2)	151
C1—H1···C*g*(*B*^{i})	0.97 (2)	2.624 (2)	3.520 (2)	153 (1)
C10—H10···C*g*(*C*^{i})	0.98 (2)	2.692 (2)	3.344 (2)	124 (1)

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

The H atoms were located from a difference Fourier map and were refined isotropically, with the exception of the H atoms attached to C22; these were geometrically placed and were treated as riding atoms, with C—H = 0.96 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

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