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4,6-Dibenzoyl-1,3-dimethylbenzene

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.055
wR factor = 0.125
Data-to-parameter ratio = 15.3

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

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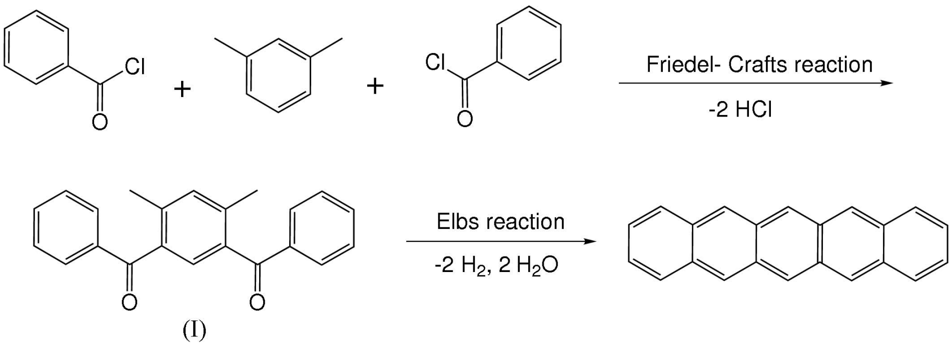
The title compound, $C_{22}H_{18}O_2$, contains three benzene rings that are not coplanar as a result of steric strain. The molecules are packed in columns along the *a* axis in the crystal structure, and classical hydrogen bonds are not found.

Comment

4,6-Dibenzoyl-1,3-dimethylbenzene, (I), is reported as the intermediate for the synthesis of pentacene (Bailey & Madoff, 1953), which is an important material for producing organic thin-film transistors (Halik *et al.*, 2002). A vigorous Friedel–Crafts reaction yields (I) from the raw materials, benzoyl chloride and *m*-xylene. Heating with copper at 623–673 K in a modified Elbs reaction converts (I) into pentacene.



The asymmetric unit of (I) consists of one molecule (Fig. 1). The three benzene rings are connected by two acyl bridges. Because of steric repulsion between the ring substituents, the molecule is not planar, with torsion angles C6—C1—C19—C15 = −153.53 (16)° and C12 –C7 –C20 –C17 = 174.57 (18)°. The bond angles and lengths are all within normal ranges. In the view along the *a* axis, the molecules of (I) are more closely packed than in other directions (Fig. 2). There are no classical hydrogen bonds in the crystal structure of (I).

Experimental

The title compound was prepared by refluxing benzoyl chloride (35 g, 0.25 mol), *m*-xylene (11 g, 0.10 mol) and anhydrous aluminium trichloride (40 g, 0.30 mol) at 413 K for 45 min. After further heating at 443 K for 30 min, the hydrochloric acid generated from the reaction was completely released. The mixture was a deep-brown coagulation when cooled to room temperature. Hydrochloric acid and sodium hydroxide were applied successively to remove the surplus anhydrous aluminium trichloride, and the dark insolubles were then decoloured by boiling in water with absorbite. The final colourless crystals (yield 8.7 g, 28%) were recrystallized several times from ethanol. Analysis calculated for $C_{22}H_{18}O_2$: H 5.77, C 84.05%;

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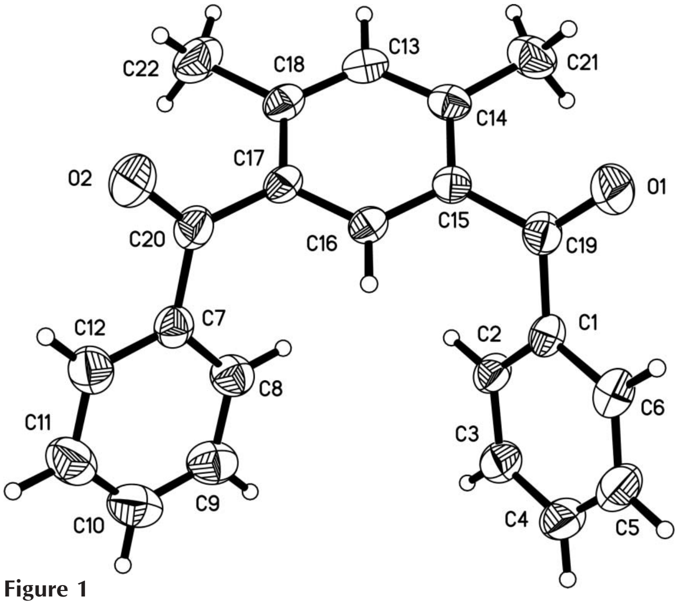

Figure 1
The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids at the 30% probability level.

found: H 5.99, C 83.36%. IR (cm^{-1}): 3058 (*w*), 1662 (*s*), 1595 (*m*), 1578 (*w*), 1446 (*m*), 1314 (*m*), 1290 (*s*), 1241 (*m*), 1175 (*w*), 1152 (*m*), 1066 (*w*), 942 (*w*), 909 (*m*), 892 (*m*), 748 (*w*), 725 (*s*), 703 (*s*), 670 (*m*). 574 (*w*). M.p. 377 K.

Crystal data

$C_{22}H_{18}O_2$
$M_r = 314.36$
Monoclinic, $P2_1/n$
$a = 9.7934$ (10) Å
$b = 8.4835$ (9) Å
$c = 20.509$ (2) Å
$\beta = 91.098$ (2)°
$V = 1703.6$ (3) Å^3
$Z = 4$
$D_x = 1.226$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 757 reflections
$\theta = 2.3$–17.8°
$\mu = 0.08$ mm^{-1}
$T = 293$ (2) K
Rod, colourless
0.32 × 0.16 × 0.14 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min} = 0.97$, $T_{\max} = 0.98$
8907 measured reflections
3340 independent reflections
2282 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\max} = 26.0°$
$h = -5 \rightarrow 12$
$k = -10 \rightarrow 10$
$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.125$
$S = 1.07$
3340 reflections
219 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 0.15P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.16$ e Å^{-3}
$\Delta\rho_{\min} = -0.13$ e Å^{-3}

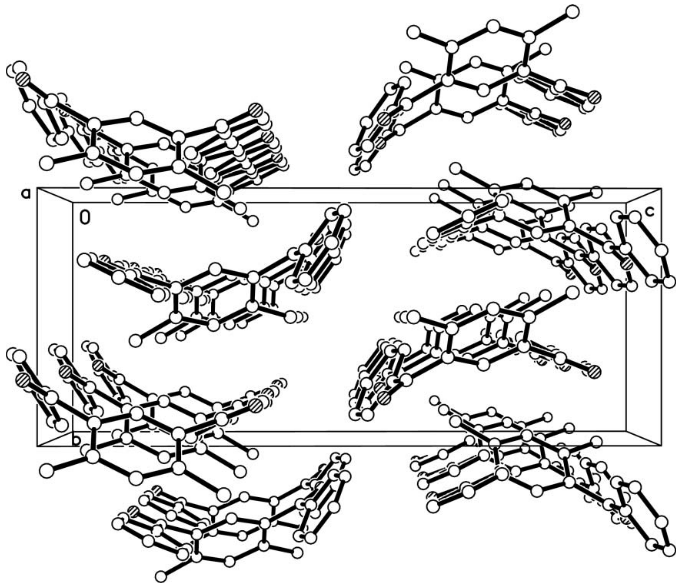

Figure 2
The crystal packing of (I), viewed down the *a* axis. H atoms have been omitted for clarity.

H atoms were positioned geometrically and were refined as riding on the parent C atoms; C—H bond distances are 0.93 Å for aromatic H atoms, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$ (C), and 0.96 Å for methyl H atoms, with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT-Plus* (Bruker, 2000); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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