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A triclinic polymorph of 1,4-dibenzoylbutane

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.036
wR factor = 0.096
Data-to-parameter ratio = 17.7

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

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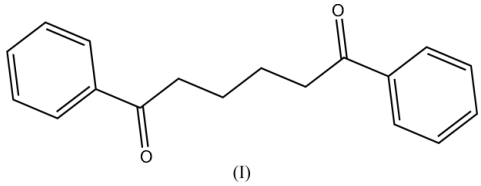
A new polymorph of 1,4-dibenzoylbutane, $C_{18}H_{18}O_2$, has been discovered. Until now only a monoclinic polymorph of the title compound was known. The newly found triclinic polymorph crystallizes in $P\overline{1}$ with half a molecule in the asymmetric unit. The two polymorphs differ slightly in the torsion angles of the central chain.

Comment

The aim of our investigations (Ton, 2004) was to synthesize a compound with two hydrazone fragments in order to examine the intramolecular hydrogen-bond interactions in the solid state. Unfortunately, we obtained crystals of 1,4-dibenzoylbutane, (I), which was one of the starting materials. The structure of (I) has already been determined at room temperature in the monoclinic space group $P2_1/n$ with $Z' = 1$ (Deguire & Brisse, 1988). We collected the data set at low temperature and obtained, surprisingly, the space group $P\overline{1}$ with $Z' = \frac{1}{2}$. To verify that the crystal structure had not undergone a phase transition upon cooling, the same crystal was investigated again, at room temperature. We still obtained the space group $P\overline{1}$. Furthermore, we determined the structure at room temperature using a crystal which had not been cooled already, and again obtained the triclinic structure. In this way, a new polymorph of (I) has been discovered.



(I)

The molecule lies on a centre of symmetry, with one-half molecule in the asymmetric unit. Whereas (I) is essentially planar (the r.m.s. deviation of all the non-H atoms is 0.019 Å), the monoclinic polymorph has an r.m.s. deviation of 0.255 Å. The two polymorphs differ mainly in the torsion angles of the central chain. A least-squares fit of all non-H atoms of the two polymorphs yields an r.m.s. deviation of 0.114 Å.

Experimental

1,4-Dibenzoylbutane was synthesized according to the method of Hünig *et al.* (1979). The intention was to use it in a further acid-catalysed reaction with benzhydrazide. After work-up, and following crystallization attempts in diethyl ether, block-shaped crystals were obtained.

Crystal data

$C_{18}H_{18}O_2$	$Z = 1$
$M_r = 266.32$	$D_x = 1.274$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 5.7482$ (12) Å	Cell parameters from 4194 reflections
$b = 7.6480$ (16) Å	$\theta = 3.8$–$27.7°$
$c = 8.2817$ (15) Å	$\mu = 0.08$ mm^{-1}
$\alpha = 95.413$ (16)°	$T = 173$ (2) K
$\beta = 105.265$ (15)°	Block, colourless
$\gamma = 95.206$ (17)°	0.52 × 0.29 × 0.27 mm
$V = 347.13$ (12) Å^3	

Data collection

Stoe IPDS-II two-circle diffractometer	1203 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.040$
Absorption correction: none	$\theta_{max} = 27.8°$
5151 measured reflections	$h = -7 \rightarrow 7$
1610 independent reflections	$k = -9 \rightarrow 9$
	$l = -10 \rightarrow 10$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.036$	$w = 1/[\sigma^2(F_o^2) + (0.0517P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.096$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.08$	$\Delta\rho_{max} = 0.29$ e Å^{-3}
1610 reflections	$\Delta\rho_{min} = -0.17$ e Å^{-3}
91 parameters	

H atoms were included with fixed individual displacement parameters [U_{iso}(H) = 1.2U_{eq}(C)] using a riding model, with aromatic C—H = 0.95 Å and methylene C—H = 0.99 Å.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

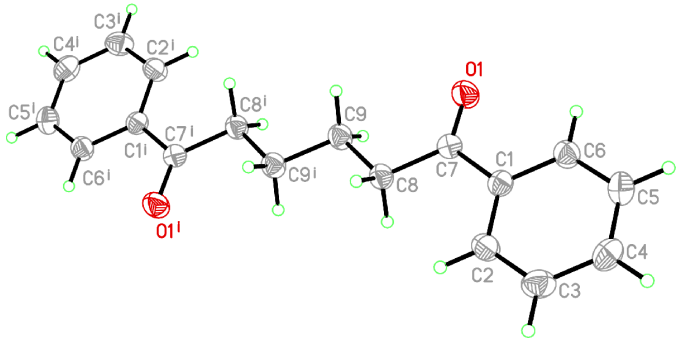


Figure 1
Perspective view of the title compound, with the atom-numbering scheme; displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (i) $2 - x$, $1 - y$, $2 - z$.]

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