

Liang-Ce Rong,* Xiao-Yue Li,
Fang Yang, Hai-Ying Wang and
Da-Qing Shi

Department of Chemistry, Xuzhou Normal
University, Xuzhou 221116, People's Republic
of China

Correspondence e-mail:
lcrong2005@yahoo.com

Key indicators

Single-crystal X-ray study
T = 294 K
Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
R factor = 0.049
wR factor = 0.142
Data-to-parameter ratio = 14.1

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

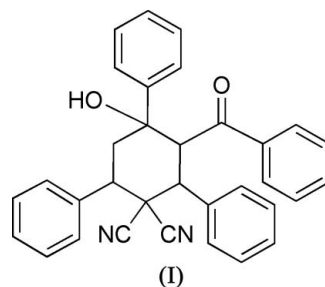
3-Benzoyl-4-hydroxy-2,4,6-triphenylcyclohexane-1,1-dicarbonitrile

The title compound, $\text{C}_{33}\text{H}_{26}\text{N}_2\text{O}_2$, was synthesized by the reaction of chalcone with malononitrile under solvent-free conditions at 353 K. X-ray analysis reveals that the cyclohexane ring adopts a chair conformation.

Received 31 March 2006
Accepted 3 April 2006

Comment

Solvent-free reactions have attracted much attention in recent years (Tanaka & Toda, 2000) and have proved to have many advantages, *viz.* reduced pollution, low costs and simplicity in processing and handling. Solid-state reactions have performed well recently (Liu *et al.*, 2001; Kaupp *et al.*, 2003; Goud *et al.*, 1995; Annunziata *et al.*, 1997). We report here the crystal structure of the title compound, (I), which was synthesized by the solvent-free reaction of chalcone with malononitrile at 353 K.



In (I), the cyclohexane ring is in a chair conformation and the phenyl rings are equatorially attached to it (Fig. 1). An intramolecular O1—H1...O2 hydrogen bond is observed in the molecular structure. The crystal packing shows that intermolecular C—H...N and C—H...O hydrogen bonds (Table 1) form a three-dimensional network (Fig. 2).

Experimental

Compound (I) was prepared by the reaction of chalcone (4 mmol) and malononitrile (2 mmol) with sodium hydroxide (0.20 g) as catalyst under solvent-free conditions. Single crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Crystal data

$\text{C}_{33}\text{H}_{26}\text{N}_2\text{O}_2$	$V = 1345.3 (4) \text{ \AA}^3$
$M_r = 482.56$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.191 \text{ Mg m}^{-3}$
$a = 10.996 (2) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 12.017 (2) \text{ \AA}$	$\mu = 0.07 \text{ mm}^{-1}$
$c = 12.109 (2) \text{ \AA}$	$T = 294 (2) \text{ K}$
$\alpha = 89.950 (4)^\circ$	Block, colourless
$\beta = 67.150 (3)^\circ$	$0.24 \times 0.16 \times 0.10 \text{ mm}$
$\gamma = 67.821 (3)^\circ$	

Data collection

Bruker SMART CCD area-detector diffractometer	6907 measured reflections
φ and ω scans	4718 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	2647 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.982$, $T_{\max} = 0.993$	$R_{\text{int}} = 0.024$
	$\theta_{\text{max}} = 25.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0573P)^2 + 0.1829P]$
$R[F^2 > 2\sigma(F^2)] = 0.049$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.142$	$(\Delta/\sigma)_{\text{max}} = 0.004$
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.14 \text{ e } \text{\AA}^{-3}$
4718 reflections	$\Delta\rho_{\text{min}} = -0.18 \text{ e } \text{\AA}^{-3}$
335 parameters	
H-atom parameters constrained	

Table 1

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O1-H1\cdots O2$	0.82	2.04	2.718 (2)	140
$C17-H17\cdots N1^i$	0.93	2.56	3.455 (5)	162
$C19-H19\cdots N2^{ii}$	0.93	2.54	3.457 (4)	168
$C21-H21\cdots O1^{iii}$	0.93	2.51	3.397 (4)	159
$C29-H29\cdots O2^{iii}$	0.93	2.57	3.441 (4)	156

Symmetry codes: (i) $x+1, y, z-1$; (ii) $-x+1, -y+2, -z+1$; (iii) $-x+1, -y+1, -z+1$.

H atoms were placed in geometrically idealized positions ($O-H = 0.82\text{\AA}$ and $C-H = 0.93-0.98\text{\AA}$) and allowed to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N})$. A search for solvent-accessible voids in the crystal structure using *PLATON* (Spek, 2003) showed a void of 58.1\AA^3 , but no significant peaks were found in the difference map.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

The authors thank the Natural Science Foundation of the Education Committee of Jiangsu Province, and the Natural Science Foundation (No. 04XLB14) of Xuzhou Normal University for financial support.

References

- Annunziata, S., Aldo, S., Margherita, R. D., Manueia, G. & Arrigo, S. (1997). *Tetrahedron Lett.* **38**, 289–290.
- Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (1999). *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Goud, B. S., Panneerselvam, K., Zacharias, D. E. & Desiraju, G. R. (1995). *J. Chem. Soc. Perkin Trans. 2*, pp. 325–330.
- Kaupp, G. M., Reza Naimi-Jamal, M. R. & Schmeyers, J. (2003). *Tetrahedron*, **59**, 3752–3760.
- Liu, H. Y., Xu, Q. H., Liang, Y. M., Chen, B. H., Liu, W. M. & Ma, Y. L. (2001). *J. Organomet. Chem.* **637–639**, 719–722.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

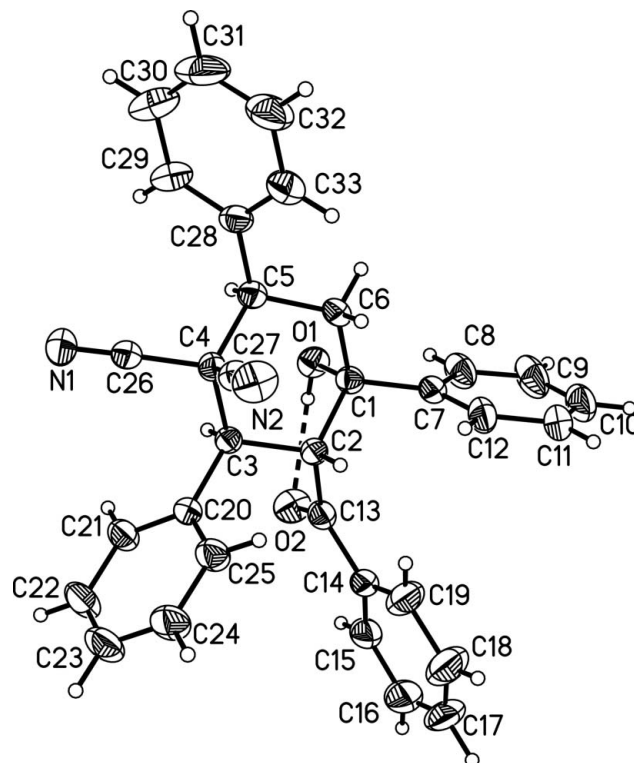


Figure 1

The molecular structure of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme. The dashed line represents an $O-H\cdots O$ hydrogen bond.

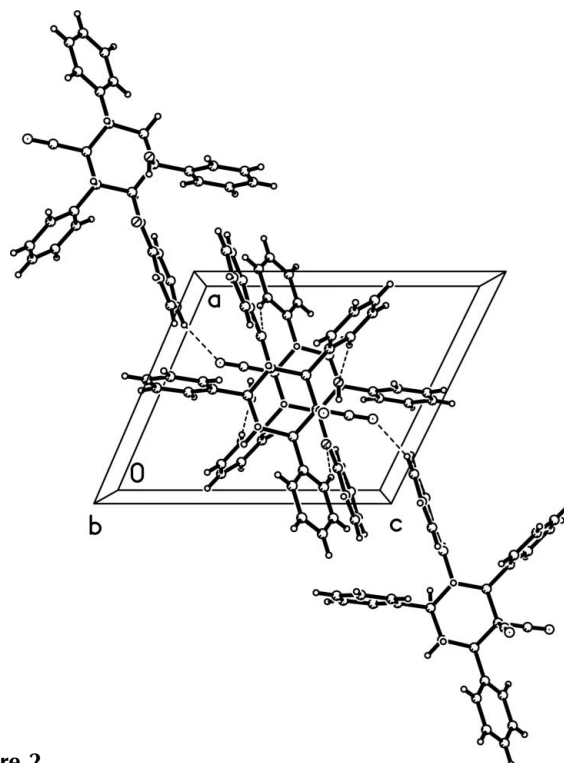


Figure 2

Part of the crystal packing of (I), showing hydrogen bonds as dashed lines.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Tanaka, K. & Toda, F. (2000). *Chem. Rev.* **100**, 1025–1074.