

## 1,5-Diphenyl-3-(2-pyridyl)pentane-1,5-dione

Xian-Qiang Huang,<sup>a\*</sup> Da-Qi Wang,<sup>a</sup> Jian-Min Dou<sup>a</sup> and Jin-Xian Wang<sup>b</sup><sup>a</sup>Department of Chemistry, Liaocheng University, Liaocheng 252059, People's Republic of China, and <sup>b</sup>Department of Chemistry, Northwest Normal University, Lanzhou 730070, People's Republic of China

Correspondence e-mail: hxqqxh2008@163.com

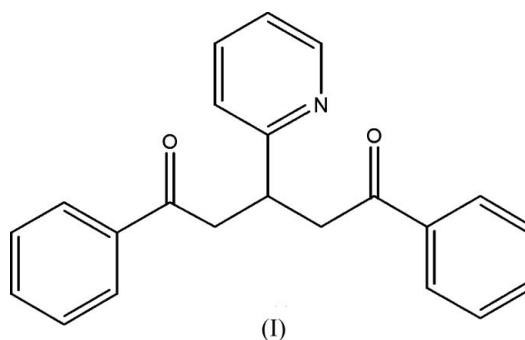
## Key indicators

Single-crystal X-ray study  
 $T = 298$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
 $R$  factor = 0.045  
 $wR$  factor = 0.128  
Data-to-parameter ratio = 14.0For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound,  $\text{C}_{22}\text{H}_{19}\text{NO}_2$ , was synthesized by the reaction of pyridine-2-carbaldehyde with acetophenone and NaOH. The bond lengths and angles show normal values. The crystal packing exhibits no significantly short intermolecular contacts.

## Comment

Organic syntheses directed towards 'green' chemistry have attracted increasing interest in recent years in view of expectations of new environmentally benign procedures saving resources and energy (Tanaka & Toda, 2000; Dittmer, 1997). The development of solvent-free reactions (DeSimone, 2002; Cave *et al.*, 2001; Tanaka, 2003) is an especially attractive goal in the context of green chemistry (Anastas & Kirchhoff, 2002). 1,5-Diketones are important synthetic intermediates and starting materials in the synthesis of many heterocyclic compounds (Hirsch & Bailey, 1978; Krohnke, 1976). It is also known that pyridine-containing compounds often exhibit strong biological activity (Keys *et al.*, 1987). In continuation of work on the synthesis of 1,5-diketones (Constable *et al.*, 1998; Fuchigami *et al.*, 1986), we present here the title new pyridine-containing compound, (I), synthesized under solvent-free conditions.



In the molecule of (I) (Fig. 1), the bond lengths and angles (Table 1) are normal and correspond to those observed in 1,3,5-triphenylpentane-1,5-diketone (Das *et al.*, 1994). The crystal packing (Fig. 2) demonstrates no significantly short intermolecular contacts.

## Experimental

Acetophenone (0.75 g, 6.25 mmol), freshly distilled pyridine-2-carbaldehyde (0.33 g, 3.125 mmol) and NaOH (0.25 g, 6.25 mmol) were mixed using a glass paddle in an open flask. The resulting product was washed with water several times to remove NaOH, and then recrystallized from ethanol (m.p. 424 K). EI-MS ( $m/e$ , %): 329

Received 21 November 2005

Accepted 1 December 2005

Online 7 December 2005

(1.62), 252 (13.14), 224(45.88), 210(11.1), 105 (100), 77(69.45);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ,  $\delta$ , p.p.m.): 8.45–7.01 (*m*, 14H), 4.24–4.17 (*m*,  $J = 6.8$  Hz, 1H), 3.63 (*dd*,  $J = 6.8$  and 16.4 Hz, 2H), 3.42 (*dd*,  $J = 6.8$  and 16.4 Hz, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ,  $\delta$ , p.p.m.): 198.56, 162.91, 149.10, 136.87, 136.37, 133.02, 128.51, 128.07, 124.12, 121.55, 43.54, 38.10; IR (KBr,  $\nu$ ,  $\text{cm}^{-1}$ ): 3003, 2889, 1680, 1589, 1246, 1209, 997, 750, 688, 540. Elemental analysis, calculated for  $\text{C}_{22}\text{H}_{19}\text{NO}_2$ : C 80.22, H 5.81, N 4.25%; found: C 80.13, H 5.87, N 4.21%.

#### Crystal data

$\text{C}_{22}\text{H}_{19}\text{NO}_2$   
 $M_r = 329.38$   
 Monoclinic,  $P2_1/c$   
 $a = 11.519$  (2) Å  
 $b = 18.162$  (3) Å  
 $c = 8.640$  (3) Å  
 $\beta = 90.213$  (2)°  
 $V = 1807.5$  (7) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.210$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 2451 reflections  
 $\theta = 2.6$ – $22.7^\circ$   
 $\mu = 0.08$  mm<sup>-1</sup>  
 $T = 298$  (2) K  
 Block, colourless  
 $0.55 \times 0.52 \times 0.47$  mm

#### Data collection

Bruker SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.959$ ,  $T_{\max} = 0.965$   
 9346 measured reflections

3189 independent reflections  
 1889 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.052$   
 $\theta_{\text{max}} = 25.0^\circ$   
 $h = -13 \rightarrow 8$   
 $k = -21 \rightarrow 20$   
 $l = -10 \rightarrow 9$

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.045$   
 $wR(F^2) = 0.128$   
 $S = 1.00$   
 3189 reflections  
 227 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2 + 0.5691P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.16$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.16$  e Å<sup>-3</sup>  
 Extinction correction: SHELXTL (Bruker, 1997)  
 Extinction coefficient: 0.049 (3)

**Table 1**

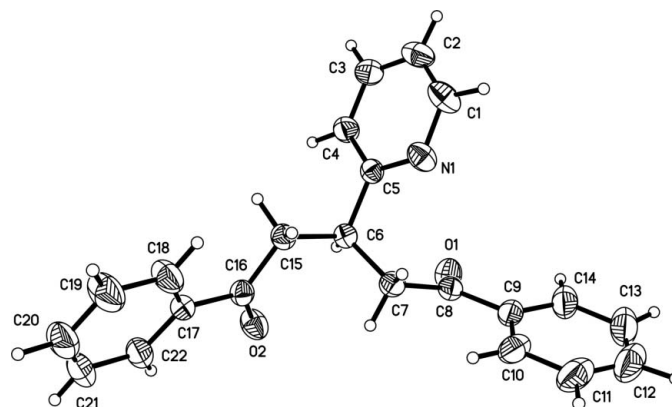
Selected geometric parameters (Å, °).

O1—C8	1.213 (2)	C6—C7	1.522 (3)
O2—C16	1.220 (2)	C6—C15	1.533 (3)
C5—C6—C7	112.54 (17)	C7—C6—C15	110.25 (17)
C5—C6—C15	110.17 (16)		

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.98 Å and  $U_{\text{iso}}(\text{H}) = 1.2$ – $1.5U_{\text{eq}}(\text{C})$ .

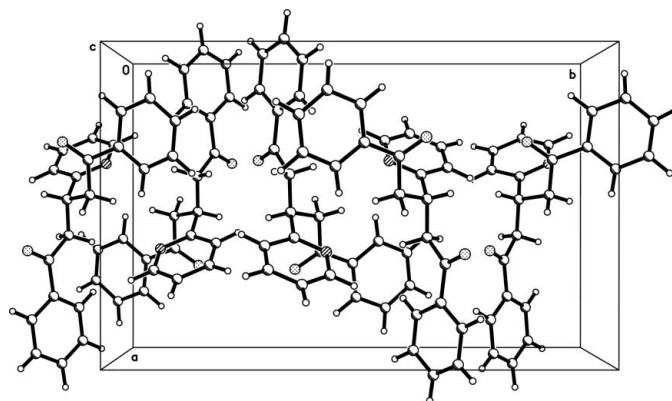
Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1997); software used to prepare material for publication: SHELXTL.

The authors acknowledge the financial support of the National Natural Science Foundation of China (grant Nos. 20371025 and 20272047).



**Figure 1**

A view of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.



**Figure 2**

A perspective view of the crystal packing of (I), along the *c* axis.

#### References

- Anastas, P. T. & Kirchoff, M. M. (2002). *Acc. Chem. Res.* **35**, 686–694.  
 Bruker (1997). SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Cave, G. W. V., Raston, C. L. & Scott, J. L. (2001). *Chem. Commun.* pp. 2159–2169.  
 Constable, E. C., Neuburger, M., Smith, D. R. & Zehnder, M. (1998). *Inorg. Chim. Acta*, **275–276**, 359–365.  
 Das, G. C., Hursthouse, M. B., Malik, K. M. A., Rahman, M. M., Rahman, M. T. & Olsson, T. (1994). *J. Chem. Crystallogr.* **24**, 511–515.  
 DeSimone, J. M. (2002). *Science*, **297**, 799–803.  
 Dittmer, D. C. (1997). *Chem. Ind.* pp. 799–784.  
 Fuchigami, T., Awata, T., Nonaka, T. & Baizer, M. M. (1986). *Bull. Chem. Soc. Jpn.* **59**, 2873–2879.  
 Hirsch, S. S. & Bailey, W. J. (1978). *J. Org. Chem.* **43**, 4090–4094.  
 Keys, L. D. & Hamilton, G. A. (1987). *J. Am. Chem. Soc.* **109**, 2156–2163.  
 Krohnke, F. (1976). *Synthesis*, pp. 1–24.  
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.  
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.  
 Siemens (1996). SMART and SAINT. Siemens Analytical X-Ray Instruments Inc., Madison, Wisconsin, USA.  
 Tanaka, K. (2003). *Solvent-free Organic Synthesis*, pp. 1–417. Weinheim: Wiley-VCH Verlag.  
 Tanaka, K. & Toda, F. (2000). *Chem. Rev.* **100**, 1025–1074.