

Canan Kazak,<sup>a\*</sup> Muhittin Aygün,<sup>b</sup> Canan Kuş,<sup>c</sup> Süheyla Özbey<sup>d</sup> and Orhan Büyükgüngör<sup>a</sup>

<sup>a</sup>Department of Physics, Ondokuz Mayıs University, TR-55139, Samsun, Turkey,

<sup>b</sup>Department of Physics, Dokuz Eylül University, Buca, TR-35150, Izmir, Turkey, <sup>c</sup>Department of Pharmaceutical Chemistry, Ankara University, 06100-Tandogan, Ankara, Turkey, and

<sup>d</sup>Department of Physics Engineering, Hacettepe University, Beytepe 06532, Ankara, Turkey

Correspondence e-mail: ckazak@samsun.omu.edu.tr

#### Key indicators

Single-crystal X-ray study

$T = 293$  K

Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å

$R$  factor = 0.052

$wR$  factor = 0.158

Data-to-parameter ratio = 15.8

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

## 5-Methyl-2-(4-methylbenzoyloxy)acetophenone

Molecules of the title compound,  $\text{C}_{17}\text{H}_{16}\text{O}_3$ , are linked *via* intermolecular  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds into infinite chains, made up of centrosymmetric bimolecular aggregates stretching along the  $a$  axis of the crystal. The 4-methylbenzene group and the carbonyl moiety are almost coplanar, and the dihedral angle between the planes of 2-(4-methylbenzoyloxy) and 5-methylacetophenone is  $78.81(5)^\circ$ .

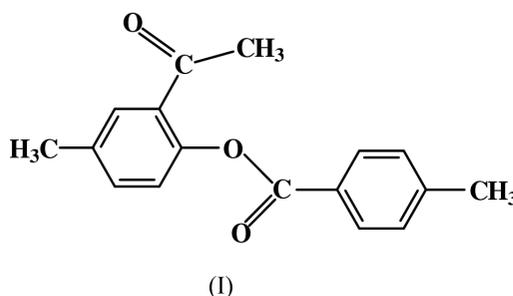
Received 11 January 2002

Accepted 26 April 2002

Online 11 May 2002

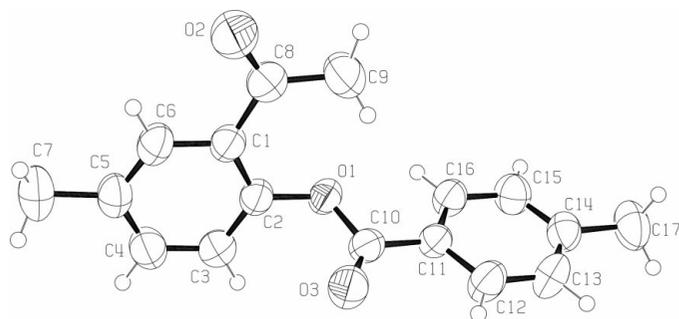
#### Comment

Acetophenone derivatives are used for the synthesis of a number of compounds containing heterocycles, such as benzofuran and benzopyran. Flavone, which has a benzopyran moiety, is an important heterocyclic system, which is present in many naturally occurring products (Mabry *et al.*, 1970) and can also be obtained synthetically. The compounds involving this heterocyclic system exhibit biological activities of various kinds, such as antiviral (Meyer *et al.*, 1991), spasmolytic (Nardi *et al.*, 1993) and antihypertensive (Wu *et al.*, 1989). 5-Methyl-2-(4-methylbenzoyloxy)acetophenone, (I), is a starting material used for the synthesis of 2-(4-methylphenyl)-6-methyl-4H-1-benzopyran-4-one (4',6-dimethylflavone) (Wurm & Nordmann, 1988).



An *ORTEP*III (Burnett & Johnson, 1996) plot of (I) is shown in Fig. 1. The  $\text{C}10=\text{O}3$  and  $\text{C}10-\text{O}1$  bond lengths are  $1.202(2)$  and  $1.361(2)$  Å, respectively, these bonds being slightly longer than the corresponding bonds in 1-(4-chlorobenzoyloxy)-2-methoxy-4-(2-propenyl)benzene [ $1.184(3)$  and  $1.348(3)$  Å, respectively; Aygün *et al.*, 1997]. The  $\text{C}8=\text{O}2$  bond length of  $1.204(2)$  Å is similar to the corresponding bond length in phenyl 2-pyridyl ketone [ $1.213(2)$  Å; Sievert *et al.*, 1998]. Other relevant bond lengths and angles are listed in Table 1.

The 5-methylacetophenone group is planar and the maximum deviations of the  $\text{C}7$  and  $\text{O}1$  atoms from its least-squares plane are  $0.033(2)$  and  $0.173(2)$  Å, respectively. The 4-methylbenzene group and the carbonyl moiety are almost



**Figure 1**  
An ORTEP drawing (Burnett & Johnson, 1996) of the title compound, showing the atomic numbering scheme. Displacement ellipsoids of non-H atoms are shown at the 50% probability level.

coplanar; the dihedral angle between their planes is 8.68 (7)°. The dihedral angle formed by the planes of the 2-(4-methylbenzoyloxy) and 5-methylacetophenone groups is 78.81 (5)°.

The crystal structure is stabilized by intermolecular C—H...O hydrogen bonds. The C3—H3...O3<sup>i</sup> [symmetry code: (i)  $-x, -y, -z$ ] hydrogen bond is responsible for formation of centrosymmetric bimolecular aggregates, and the C4—H4...O2<sup>ii</sup> [symmetry code: (ii)  $x-1, y, z$ ] hydrogen bond links these aggregates into infinite chains stretching along the *a* axis of the crystal. The geometric parameters of the hydrogen bonds are given in Table 2.

## Experimental

*p*-Tolylchloride (5.845 g, 0.0378 mol, 5 ml) was added to a solution of 2-hydroxy-5-methylacetophenone (Aktiebolag, 1965) (5.67 g, 0.0378 mol) in pyridine (8 ml) and heated for 0.5 h at 353 K. The mixture was poured into water, acidified with HCl and extracted with CHCl<sub>3</sub>. The CHCl<sub>3</sub> layer was washed with water, dried over MgSO<sub>4</sub> and evaporated. Crystallization of the residue from EtOH gave 7.5 g of the title compound.

### Crystal data

|  |  |
|--|--|
| C <sub>17</sub> H <sub>16</sub> O <sub>3</sub> | <i>Z</i> = 2                                   |
| <i>M<sub>r</sub></i> = 268.30                  | <i>D<sub>x</sub></i> = 1.24 Mg m <sup>-3</sup> |
| Triclinic, <i>P</i> $\bar{1}$                  | Cu <i>K</i> α radiation                        |
| <i>a</i> = 7.5424 (6) Å                        | Cell parameters from 25 reflections            |
| <i>b</i> = 9.5834 (8) Å                        | <i>θ</i> = 21.5–42.7°                          |
| <i>c</i> = 10.7427 (8) Å                       | <i>μ</i> = 0.68 mm <sup>-1</sup>               |
| <i>α</i> = 69.671 (7)°                         | <i>T</i> = 293 (2) K                           |
| <i>β</i> = 80.402 (9)°                         | Prismatic, colorless                           |
| <i>γ</i> = 87.077 (8)°                         | 0.36 × 0.21 × 0.15 mm                          |
| <i>V</i> = 717.9 (1) Å <sup>3</sup>            |  |

### Data collection

|  |   |
|--|---|
| Enraf–Nonius CAD-4 diffractometer                                | 2228 reflections with <i>I</i> > 2σ( <i>I</i> ) |
| ω/2θ scans   | <i>θ</i> <sub>max</sub> = 74.2°                 |
| Absorption correction: ψ scan (North <i>et al.</i> , 1968)       | <i>h</i> = 0 → 9                                |
| <i>T</i> <sub>min</sub> = 0.752, <i>T</i> <sub>max</sub> = 0.905 | <i>k</i> = -11 → 11                             |
| 2926 measured reflections  | <i>l</i> = -12 → 13                             |
| 2926 independent reflections                                     | 3 standard reflections                          |
|  | frequency: 120 min                              |
|  | intensity decay: 2%                             |

### Refinement

|                                     |   |
|-------------------------------------|---|
| Refinement on <i>F</i> <sup>2</sup> | $w = 1/[\sigma^2(F_o^2) + (0.0848P)^2 + 0.0632P]$ |
| $R[F^2 > 2\sigma(F^2)] = 0.052$     | where $P = (F_o^2 + 2F_c^2)/3$                    |
| $wR(F^2) = 0.158$                   | (Δ/σ) <sub>max</sub> = 0.005                      |
| <i>S</i> = 1.09                     | Δρ <sub>max</sub> = 0.22 e Å <sup>-3</sup>        |
| 2926 reflections                    | Δρ <sub>min</sub> = -0.22 e Å <sup>-3</sup>       |
| 185 parameters                      | Extinction correction: SHELXL97                   |
| H-atom parameters constrained       | Extinction coefficient: 0.0061 (15)               |

**Table 1**

Selected geometric parameters (Å, °).

|          |           |             |           |
|----------|-----------|-------------|-----------|
| O1—C10   | 1.360 (2) | C1—C8       | 1.502 (2) |
| O1—C2    | 1.406 (2) | C8—C9       | 1.489 (3) |
| O2—C8    | 1.204 (2) | C10—C11     | 1.474 (2) |
| O3—C10   | 1.202 (2) | C14—C17     | 1.507 (2) |
| C7—C5    | 1.508 (2) |             |           |
| C6—C5—C7 | 121.5 (2) | O2—C8—C1    | 119.1 (2) |
| C3—C2—O1 | 117.6 (1) | O3—C10—O1   | 122.2 (1) |
| C1—C2—O1 | 120.4 (1) | O3—C10—C11  | 125.6 (1) |
| C2—C1—C6 | 116.6 (2) | O1—C10—C11  | 112.1 (1) |
| C2—C1—C8 | 126.2 (2) | C16—C11—C10 | 123.0 (1) |
| O2—C8—C9 | 118.6 (2) | C13—C14—C17 | 121.2 (2) |

**Table 2**

Hydrogen-bonding geometry (Å, °).

| <i>D</i> —H... <i>A</i>  | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|--------------------------|-------------|---------------|-----------------------|-------------------------|
| C4—H4...O3 <sup>i</sup>  | 0.93        | 2.54          | 3.442 (3)             | 163                     |
| C3—H3...O2 <sup>ii</sup> | 0.93        | 2.55          | 3.404 (2)             | 153                     |

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

H atoms were placed in positions calculated on stereochemical grounds and included in the refinement in the riding-motion approximation. Their *U*<sub>iso</sub> values were constrained to be 1.2 times *U*<sub>eq</sub> of the carrier atom (1.5*U*<sub>eq</sub> in the case of the methyl H atoms).

Data collection: CAD-4 EXPRESS (Enraf–Nonius, 1993); cell refinement: CAD-4 EXPRESS; data reduction: MolEN (Fair, 1990); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP III (Burnett & Johnson, 1996); software used to prepare material for publication: SHELXL97.

The authors acknowledge the use of the CAD-4 diffractometer (purchased under grant DPT/TBAG1 of the Scientific and Technical Research Council of Turkey) of the Physics Engineering Department, Hacettepe University, Turkey.

## References

- Aktiebolag, H. (1965). *Apotekare Paul Nordstroms Fabriker. Synthesis of benzofuran derivatives*. Neth. Patent 6 413 996; *Chem. Abstr.* (1965). **63**, 18043.
- Aygün, M., Işık, Ş., Şaşmaz, S., Tahir, M. N., Erdönmez, A. & Büyükgüngör, O. (1997). *Acta Cryst.* **C53**, 897–899.
- Burnett, M. N. & Johnson, C. K. (1996). ORTEP III. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
- Enraf–Nonius (1993). CAD-4 EXPRESS. Version 1.1. Enraf–Nonius, Delft, The Netherlands.
- Fair, C. K. (1990). MolEN. Enraf–Nonius, Delft, The Netherlands.
- Mabry, T. J., Markham, K. R. & Thomas, M. B. (1970). *The Systematic Identification of Flavanoids*. Berlin: Springer Verlag.
- Meyer, N. D., Haemers, A., Mishra, L., Pandey, H. K., Pieters, L. A. C., Berghe, D. A. V. & Vlietinck, A. J. (1991). *J. Med. Chem.* **34**, 736–746.

- Nardi, D., Leonardi, A., Pennini, R., Tajana, A., Cazzulani, P. & Testa, R. (1993). *Arzneim-Forsch./Drug Res.* **43**, 28–34.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–354.
- Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
- Sievert, M., Dienelt, R. & Bock, H. (1998). *Acta Cryst.* **C54**, 674–676.
- Wu, E. S. C., Cole, T. E., Davidson, T. A., Dailey, M. A., Doring, K. G., Fedorchuk, Loch, J. T., Thomas, T. L., Biosser, J. C., Borrelli, A. R., Kinsolving, C. R., Parker, R. B., Strand, J. C. & Watkins, B. E. (1989). *J. Med. Chem.* **32**, 183–192.
- Wurm, G. & Nordmann, M. (1988). *Arch. Pharm. (Weinheim)*, **321**, 555–558.