

## 3-(3-Oxo-1,3-dihydroisobenzofuran-1-ylidene)pentane-2,4-dione

Jaime Portilla,<sup>a</sup> Jairo Quiroga,<sup>a</sup> Justo Cobo,<sup>b</sup> John N. Low<sup>c</sup> and Christopher Glidewell<sup>d\*</sup>

<sup>a</sup>Grupo de Investigación de Compuestos, Heterocíclicos, Departamento de Química, Universidad de Valle, AA 25360, Colombia, <sup>b</sup>Departamento de Química Inorgánica y Orgánica, Universidad de Jaén, 23071 Jaén, Spain, <sup>c</sup>Department of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB24 3UE, Scotland, and <sup>d</sup>School of Chemistry, University of St Andrews, Fife KY16 9ST, Scotland  
Correspondence e-mail: cg@st-andrews.ac.uk

Received 9 April 2007

Accepted 10 April 2007

Online 11 May 2007

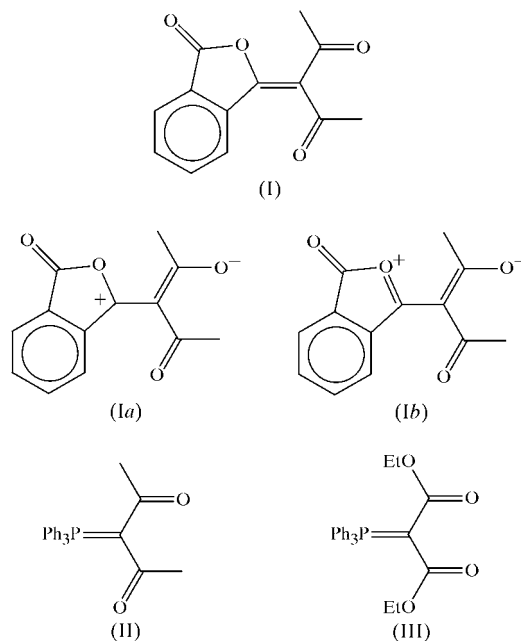
In the title compound, C<sub>13</sub>H<sub>10</sub>O<sub>4</sub>, prepared by the condensation reaction between phthalic anhydride and pentane-2,4-dione, one of the acetyl groups in the pentane-2,4-dione fragment is almost coplanar with the isobenzofuran ring system, while the other group is twisted out of this plane by almost 80°. The corresponding C—C and C—O distances in the two acetyl units are consistent with dipolar delocalization into the coplanar unit only. There is no supramolecular aggregation of the molecules, but a number of rather short intermolecular contacts of C—H...O, O...O and O...C types occur.

## Comment

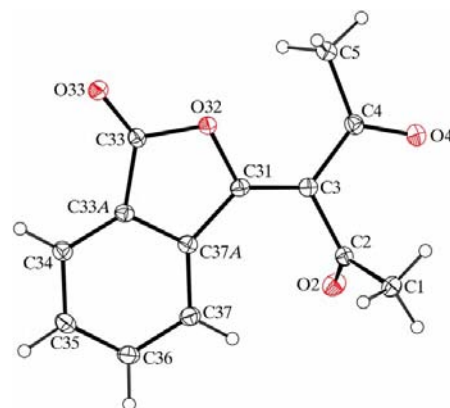
As part of a programme for the synthesis of new fused heterocyclic systems of potential biological interest, we have evaluated the use of 3-(3-oxo-1,3-dihydroisobenzofuran-1-ylidene)pentane-2,4-dione, (I), as a new  $\alpha,\beta$ -unsaturated ketone derivative. We report here the structure of (I), which was prepared by condensation of phthalic anhydride and pentane-2,4-dione.

The bond lengths within the carbocyclic ring span the very narrow range 1.387 (2)–1.394 (2) Å, indicative of unperturbed aromatic delocalization; in the heterocyclic ring, the C31—C37A and C33—C33A distances are very similar (Table 1), as are the C31—O32 and C33—O32 distances. However, in the pentane-2,4-dione fragment, the two C=O distances are significantly different, as are the C2—C3 and C3—C4 distances. These differences in bond lengths are associated with the conformations adopted by the two acetyl groups. Atoms C2, C3, C4, O4 and C5 are almost coplanar with the isobenzofuran unit, as shown by the key torsion angles (Table 1). However, the C1/C2/O2 acetyl group is twisted out of this plane by almost 80° (Table 1 and Fig. 1); the dihedral angles between the C1—C3/O2 and C2/C3/C4/C31/O32 mean planes is 79.7 (2)°. The bond distances in the pentane-2,4-

dione fragment are thus consistent with the dipolar form (Ia) (see scheme) as a significant contributor to the overall molecular electronic structure, but the close similarity of the C—O distances in the furan ring effectively rules out any contribution from dipolar form (Ib).



There are two direction-specific intermolecular interactions of possible significance in the structure of (I). The first is a C—H...O interaction (Table 2), where the H...O distance is probably too long and the C—H...O angle is probably too small for this contact to be described as a significant hydrogen bond. Since both the C and the O atoms involved are located in the isobenzofuran ring, it is not possible to find in this interaction any explanation for the differing conformations of the two acetyl units. The second interaction involves rather short contacts between acetyl atom O2 in the molecule at (x, y, z) and the sequence of atoms C31, O32 and C33 in the molecule at (−x, 1 − y, 1 − z); the interatomic distances to C31, O32 and C33 are, respectively, 2.994 (2), 2.831 (2) and 3.005 (2) Å, associated with C—H...X angles of 126.5 (2),



**Figure 1**  
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

153.6 (2) and 144.0 (2)° for  $X = \text{C31}, \text{O32}$  and  $\text{C33}$ , respectively. The expected polarization of the O and C atoms involved suggests that the  $\text{O2} \cdots \text{O32}^{\text{ii}}$  interaction [symmetry code: (ii)  $-x, -y + 1, -z + 1$ ] is repulsive, while the  $\text{O2} \cdots \text{C31}^{\text{ii}}$  and  $\text{O2} \cdots \text{C33}^{\text{ii}}$  interactions are both attractive. As with the intermolecular  $\text{C}-\text{H} \cdots \text{O}$  contact, it is not easy to see in these  $\text{O} \cdots \text{O}$  and  $\text{O} \cdots \text{C}$  contacts any simple explanation for the molecular conformation of (I).

In contrast to the molecular conformation adopted by (I), in 3-(triphenylphosphoranylidene)pentane-2,4-dione, (II), the entire pentane-2,4-dione unit is close to planarity in the *syn-anti* conformation, with only modest differences between the corresponding bond distances in the two acetyl units (Castañeda *et al.*, 2005). However, in the closely-related compound diethyl 2-(triphenylphosphoranylidene)malonate, (III), both ester units are twisted out of the plane of the central  $\text{PC}_3$  unit (Castañeda *et al.*, 2005).

## Experimental

To a solution of phthalic anhydride (20 mmol) and pentane-2,4-dione (20 mmol) in acetic anhydride (11.3 ml) at 298 K, triethylamine (40 mmol) was added dropwise and the mixture stirred for 30 min. The reaction was quenched by the addition of aqueous hydrochloric acid (45 ml of 1 mol  $\text{dm}^{-3}$  solution). The resulting solid was collected by filtration, and washed with diethyl ether (two aliquots of 25 ml) and then with hexane (two aliquots of 25 ml) to give (I) as colourless crystals, which were suitable for single-crystal X-ray diffraction (yield 90%; m.p. 406–408 K). MS (70 eV)  $m/z$  (%): 230 (5,  $M^+$ ), 187 (39), 172 (100), 104 (39), 89 (74), 76 (38), 43 (66), 42 (96), 15 (44).

### Crystal data

$\text{C}_{13}\text{H}_{10}\text{O}_4$	$V = 1056.90$ (12) $\text{\AA}^3$
$M_r = 230.21$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 8.2779$ (3) $\text{\AA}$	$\mu = 0.11$ $\text{mm}^{-1}$
$b = 8.4719$ (6) $\text{\AA}$	$T = 120$ (2) K
$c = 15.1413$ (12) $\text{\AA}$	$0.52 \times 0.31 \times 0.08$ mm
$\beta = 95.534$ (6)°	

### Data collection

Bruker–Nonius KappaCCD diffractometer	25118 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	2426 independent reflections
$T_{\text{min}} = 0.964$ , $T_{\text{max}} = 0.991$	1813 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.043$

**Table 1**

Selected geometric parameters ( $\text{\AA}$ , °).

$\text{C2}-\text{O2}$	1.211 (2)	$\text{C31}-\text{C37A}$	1.470 (2)
$\text{C4}-\text{O4}$	1.225 (2)	$\text{C33}-\text{C33A}$	1.464 (2)
$\text{C2}-\text{C3}$	1.518 (2)	$\text{O32}-\text{C31}$	1.389 (2)
$\text{C3}-\text{C4}$	1.489 (2)	$\text{O32}-\text{C33}$	1.387 (2)
$\text{C3}-\text{C31}$	1.348 (3)	$\text{C33}-\text{O33}$	1.204 (2)
$\text{C2}-\text{C3}-\text{C31}-\text{O32}$	175.40 (14)	$\text{C4}-\text{C3}-\text{C31}-\text{O32}$	-6.2 (3)
$\text{O2}-\text{C2}-\text{C3}-\text{C31}$	77.9 (2)	$\text{O4}-\text{C4}-\text{C3}-\text{C31}$	175.99 (17)
$\text{C1}-\text{C2}-\text{C3}-\text{C31}$	-101.57 (19)	$\text{C5}-\text{C4}-\text{C3}-\text{C31}$	-6.1 (3)

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$	156 parameters
$wR(F^2) = 0.139$	H-atom parameters constrained
$S = 1.08$	$\Delta\rho_{\text{max}} = 0.27$ e $\text{\AA}^{-3}$
2426 reflections	$\Delta\rho_{\text{min}} = -0.24$ e $\text{\AA}^{-3}$

**Table 2**

Parameters for the short intermolecular  $\text{C}-\text{H} \cdots \text{O}$  contact ( $\text{\AA}$ , °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{C35}-\text{H35} \cdots \text{O33}^{\text{i}}$	0.95	2.57	3.256 (2)	130

Symmetry code: (i)  $x, -y + \frac{1}{2}, z - \frac{1}{2}$ .

The space group  $P2_1/c$  was uniquely assigned from the systematic absences. All H atoms were located in difference maps and then treated as riding atoms in geometrically idealized positions, with  $\text{C}-\text{H}$  distances of 0.95 (aromatic) or 0.98  $\text{\AA}$  (methyl), and with  $U_{\text{iso}}(\text{H}) = kU_{\text{eq}}(\text{C})$ , where  $k = 1.2$  for the aromatic ring and  $k = 1.5$  for the methyl groups.

Data collection: COLLECT (Hooft, 1999); cell refinement: DIRAX/LSQ (Duisenberg *et al.*, 2000); data reduction: EVALCCD (Duisenberg *et al.*, 2003); program(s) used to solve structure: SIR2004 (Burla *et al.*, 2005) and WinGX (Farrugia, 1999); program(s) used to refine structure: OSCAIL (McArdle, 2003) and SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97 and PRPKAPPA (Ferguson, 1999).

The authors thank ‘Servicios Técnicos de Investigación of Universidad de Jaén’ and the staff for data collection. JC thanks the Consejería de Innovación, Ciencia y Empresa (Junta de Andalucía, Spain) and the Universidad de Jaén for financial support. JP and JQ thank COLCIENCIAS and UNIVALLE (Universidad del Valle, Colombia) for financial support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK3122). Services for accessing these data are described at the back of the journal.

## References

- Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. (2005). *J. Appl. Cryst.* **38**, 381–388.
- Castañeda, F., Aliaga, C., Bunton, C. A., Garland, M. T. & Baggio, R. (2005). *Acta Cryst.* **C61**, o496–o499.
- Duisenberg, A. J. M., Hooft, R. W. W., Schreurs, A. M. M. & Kroon, J. (2000). *J. Appl. Cryst.* **33**, 893–898.
- Duisenberg, A. J. M., Kroon-Batenburg, L. M. J. & Schreurs, A. M. M. (2003). *J. Appl. Cryst.* **36**, 220–229.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Ferguson, G. (1999). PRPKAPPA. University of Guelph, Canada.
- Hooft, R. W. W. (1999). COLLECT. Nonius BV, Delft, The Netherlands.
- McArdle, P. (2003). OSCAIL for Windows. Version 10. Crystallography Centre, Chemistry Department, NUI Galway, Ireland.
- Sheldrick, G. M. (1997). SHELXL97. University of Göttingen, Germany.
- Sheldrick, G. M. (2003). SADABS. Version 2.10. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.