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1,5-Bis(4-bromophenyl)-3-(3-nitrophenyl)-pentane-1,5-dione

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In the the title compound, $C_{23}H_{17}Br_2NO_4$, the two bromophenyl rings are almost coplanar, and perpendicular to the nitrophenyl ring. The carbonyl groups are coplanar with their adjacent bromophenyl rings.

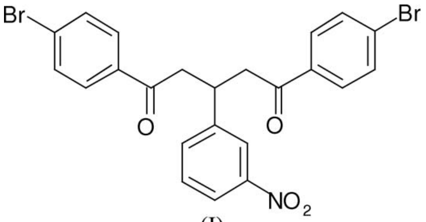
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
R factor = 0.067
wR factor = 0.129
Data-to-parameter ratio = 14.4

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

Comment

Chalcones are finding applications as organic non-linear optical materials (NLO) due to their good SHG conversion efficiencies (Sarojini *et al.*, 2006). Recently, we have reported the crystal structures of the substituted chalcones 1,5-bis(4-chlorophenyl)penta-1,4-dien-3-one (Butcher *et al.*, 2006) and 1,5-bis(3-bromo-2-thienyl)-3-(3-nitrophenyl)pentane-1,5-dione (Yathirajan *et al.*, 2006). The related structures of 1,5-bis(4-chlorophenyl)-3-(2-chloroquinolin-3-yl)pentane-1,5-dione (Insuasty *et al.*, 2006), 1,5-(4-dichlorophenyl)-3-(2,5-dimethoxyphenyl) pentane-1,5-dione (Teh *et al.*, 2006), 3-(2-furyl)-1,5-bis(4-methylphenyl)pentane-1,5-dione (Huang *et al.*, 2006), 1,5-bis(4-chlorophenyl)-3-(4-pyridyl)pentane-1,5-dione (Qiu, Liu & Zhu, 2006) and 3-(3-chlorophenyl)-1,5-bis(4-nitrophenyl)pentane-1,5-dione (Qiu, Yang *et al.*, 2006) have been reported. We now describe the synthesis and crystal structure of the title compound, (I) (Fig. 1). It crystallises in a centrosymmetric space group, therefore its SHG response is zero.



(I)

The bond lengths and angles in (I) can be regarded as normal (Cambridge Structural Database, Version 5.27, November 2005 updated August 2006; *MOGUL* Version 1.1; Allen, 2002). The two *p*-bromophenyl rings are coplanar [dihedral angle = 4.2 (3)°; r.m.s. deviation for the 12 ring atoms = 0.031 Å] and more or less perpendicular to the *m*-nitrophenyl ring [dihedral angles: 84.78 (15) and 84.78 (15)° for the rings containing Br1 and Br2, respectively]. The two carbonyl groups are coplanar with their adjacent bromophenyl rings (Table 1).

Experimental

4-Bromoacetophenone (19.1 g, 0.096 mol) in methanol (50 ml) was mixed with 3-nitrobenzaldehyde (7.2 g, 0.048 mol) and the mixture was treated with 10 ml of 30% aqueous potassium hydroxide solution at 278 K. The reaction mixture was then brought to 300 K and stirred for 6 h. The precipitated solid was filtered and washed with water, dried and recrytallized from acetone to yield orange–brown crystals of (I) (yield: 65%; m.p. 383–85 K). Analysis for $C_{23}H_{17}Br_2NO_4$ found (calculated): C 51.09 (52.00), H 3.18 (3.23), N 2.60% (2.64%).

Crystal data

$C_{23}H_{17}Br_2NO_4$
$M_r = 531.20$
Orthorhombic, *Pbca*
$a = 21.3111\ (10)$ Å
$b = 9.3462\ (5)$ Å
$c = 22.3385\ (16)$ Å
$V = 4449.3\ (4)$ Å^3

$Z = 8$
$D_x = 1.586$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 3.67$ mm^{-1}
$T = 293\ (2)$ K
Block, light orange–brown
0.25 × 0.21 × 0.13 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
$T_{\min} = 0.430$, $T_{\max} = 0.637$

37736 measured reflections
3916 independent reflections
3049 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.085$
$\theta_{\max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.067$
$wR(F^2) = 0.129$
$S = 1.18$
3916 reflections
272 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0312P)^2 + 8.8217P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.67$ e Å^{-3}
$\Delta\rho_{\min} = -0.55$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00139 (19)

Table 1
Selected torsion angles (°).

O1—C1—C11—C12	−2.4 (8)	O2—C5—C21—C22	−0.7 (8)

The H atoms were found in a difference map, repositioned in idealized locations (C—H = 0.93–0.98Å) and refined as riding, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PLATON*.

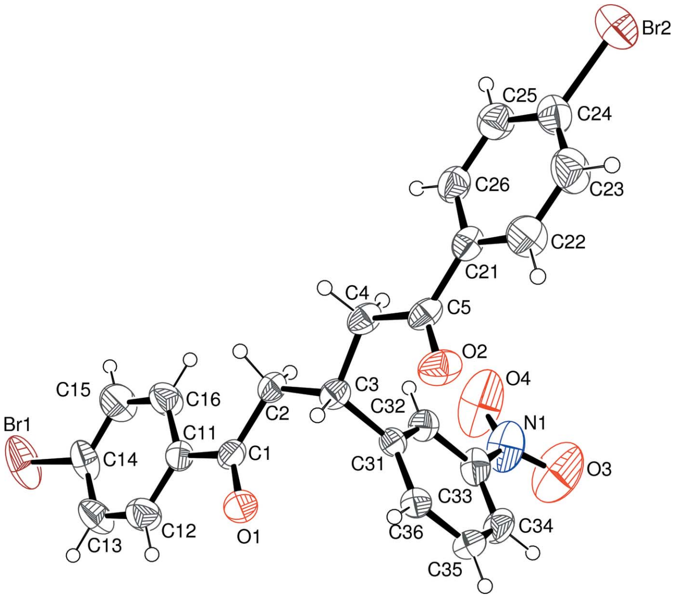


Figure 1
The molecular structure of (I), showing 40% displacement ellipsoids (arbitrary spheres for the H atoms).

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