

2,3-Dibromo-1-(2,4-dichlorophenyl)-3-(4,5-dimethoxy-2-nitrophenyl)propan-1-one

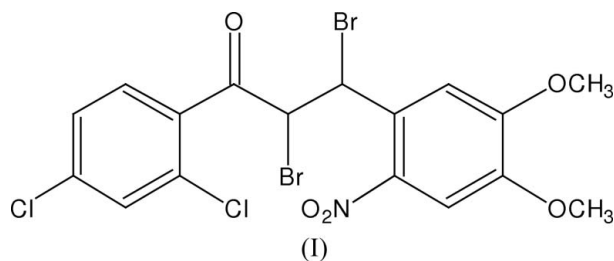
H. S. Yathirajan,^a Anil N. Mayekar,^a B. Narayana,^a B. K. Sarojini^b and Michael Bolte^{c*}^aDepartment of Studies in Chemistry, University of Mysore, Manasagangothri, Mysore 570 006, India, ^bDepartment of Chemistry, P. A. College of Engineering, Nadupadavu, Mangalore 574 153, India, and ^cInstitut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Max-von-Laue-Strasse 7, 60438 Frankfurt/Main, GermanyCorrespondence e-mail:
bolte@chemie.uni-frankfurt.de

Key indicators

Single-crystal X-ray study
 $T = 173$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.039
 wR factor = 0.093
Data-to-parameter ratio = 16.1For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The title compound, $\text{C}_{17}\text{H}_{13}\text{Br}_2\text{Cl}_2\text{NO}_5$, crystallizes with two similar molecules in the asymmetric unit. The Br atoms are *trans* to each other.

Comment

For a structurally simple group of compounds, chalcones display an impressive array of biological activities, among which antimalarial (Liu *et al.*, 2003), antiprotozoal (Nielson *et al.*, 1998), nitric oxide inhibition (Rajas *et al.*, 2002) and anti-cancer activities (Dinkova-Kostova *et al.*, 1998) have been reported in the literature. Among several organic compounds reported for non-linear optical (NLO) properties, chalcone derivatives are notable materials for their excellent blue light transmittance and good crystallizability. They provide a necessary configuration to show NLO properties, with two planar rings connected through a conjugated double bond (Goto *et al.*, 1991; Uchida *et al.*, 1998; Tam *et al.*, 1989; Indira *et al.*, 2002; Sarojini *et al.*, 2006). The substitution of a bromo group on either of the phenyl rings greatly influences the non-centrosymmetric crystal packing. The bromo group can obviously improve the molecular first-order hyperpolarizabilities and can effectively reduce dipole–dipole interactions between the molecules. Chalcone derivatives usually have a lower melting temperature, which can be a drawback when we use these crystals in optical instruments. Chalcone dibromides usually have higher melting points and are thermally stable. Only a few structures of these compounds have been reported (Butcher *et al.*, 2006; Yathirajan *et al.*, 2007; Harrison *et al.*, 2005). In continuation of our studies of chalcones and their derivatives (Yathirajan *et al.*, 2006, and references therein), the title chalcone dibromide, (I), was prepared by the bromination of the chalcone and its crystal structure is reported.



The asymmetric unit contains two molecules; a perspective view is shown in Fig. 1. Bond lengths and angles can be regarded as normal [Cambridge Crystallographic Database, Version 5.28, November 2006, updated January 2007 (Allen, 2002); *Mogul*, Version 1.1 (Bruno *et al.*, 2004)]. The two

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molecules of (I) in the asymmetric unit are very similar: a least-squares fit of all non-H atoms gives an r.m.s. deviation of 0.659 Å. The Br atoms are *trans* to each other. The nitro groups are slightly twisted out of the plane of the aromatic ring to which they are attached (Table 1).

Experimental

(2*E*)-1-(2,4-Dichlorophenyl)-3-(4,5-dimethoxy-2-nitrophenyl)prop-2-en-1-one (3.82 g, 0.01 mol) was treated with bromine in acetic acid (30%) until the orange colour of the solution persisted. After stirring for half an hour, the contents were poured on to crushed ice. The resulting solid mass was collected by filtration. The compound was dried and recrystallized from ethanol. Crystals of (I) suitable for structure determination were obtained from a solution in an acetone–toluene mixture (1:1 *v/v*) by slow evaporation (yield 85%; m.p. 441–443 K). Analysis for C₁₇H₁₃Br₂Cl₂NO₅: found (calculated): C 37.53 (37.67), H 2.38 (2.42), N 2.54% (2.58%).

Crystal data

C ₁₇ H ₁₃ Br ₂ Cl ₂ NO ₅	$V = 7731.6 (4) \text{ \AA}^3$
$M_r = 542.00$	$Z = 16$
Orthorhombic, <i>Pbcn</i>	Mo $K\alpha$ radiation
$a = 13.7847 (4) \text{ \AA}$	$\mu = 4.50 \text{ mm}^{-1}$
$b = 18.7740 (5) \text{ \AA}$	$T = 173 (2) \text{ K}$
$c = 29.8756 (10) \text{ \AA}$	$0.35 \times 0.32 \times 0.22 \text{ mm}$

Data collection

Stoe IPDSII two-circle diffractometer	114175 measured reflections
Absorption correction: multi-scan (<i>MULABS</i> ; Spek, 2003; Blessing, 1995)	7902 independent reflections
$T_{\min} = 0.232$, $T_{\max} = 0.388$	6553 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.090$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$	491 parameters
$wR(F^2) = 0.093$	H-atom parameters constrained
$S = 1.04$	$\Delta\rho_{\text{max}} = 1.05 \text{ e \AA}^{-3}$
7902 reflections	$\Delta\rho_{\text{min}} = -0.99 \text{ e \AA}^{-3}$

Table 1

Selected torsion angles (°).

O1–N1–C12–C11	–40.5 (5)	O2A–N1A–C12A–C11A	–27.1 (6)
O2–N1–C12–C13	–37.8 (5)	O1A–N1A–C12A–C13A	–25.8 (6)

H atoms were found in a difference map but were refined using a riding model, with C–H ranging from 0.95 to 1.00 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$. The methyl groups were allowed to rotate but not to tip. The highest residual electron density peak is located 1.05 Å from atom Br1A.

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: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

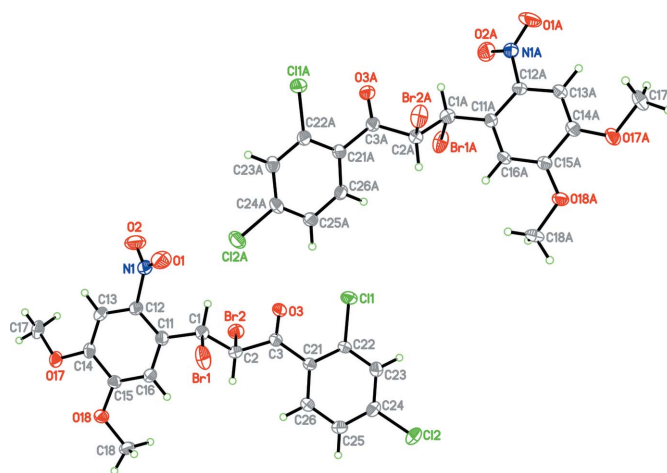


Figure 1

The asymmetric unit of (I), with the atom numbering. Displacement ellipsoids are drawn at the 50% probability level.

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