

**(8-Bromonaphthalen-1-yl)methyl 8-bromo-1-naphthoate**Igor Novak,<sup>a\*</sup> Wei Li<sup>b</sup> and  
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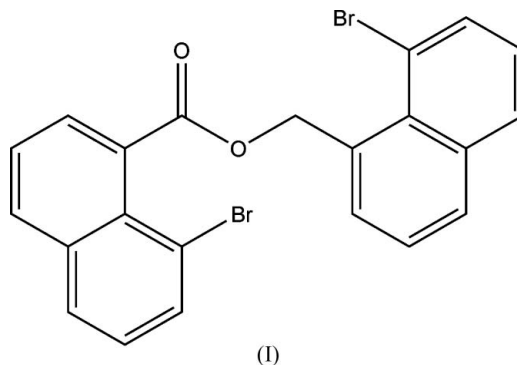
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**Key indicators**Single-crystal X-ray study  
 $T = 223$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.015$  Å  
 $R$  factor = 0.071  
 $wR$  factor = 0.236  
Data-to-parameter ratio = 13.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The structure of the title compound,  $\text{C}_{22}\text{H}_{14}\text{Br}_2\text{O}_2$  reveals the *anti* conformation of the two naphthalene rings. The plane of the ester group is almost perpendicular to the planes of the two aromatic rings.

**Comment**

Aryl esters may undergo photoinduced decarboxylation (Gu *et al.* 2001) rather than photo-Fries rearrangements. These decarboxylation processes were shown to be influenced by molecular conformations, but very little is known about the conformations of aryl esters in singlet ground states. Therefore, we determined the crystal structure of the title aryl ester. The geometry of the ester group (*i.e.* CO bond lengths and OCC bond angles) in the title compound, (I) (Fig. 1), is almost identical to the geometry of the carboxyl group in 1-naphthoic acid (Fitzgerald *et al.* 1993). In 1-naphthoic acid, the dihedral angle between the naphthalene ring system and the carboxyl group is  $8.4(3)^\circ$ ; however, in (I), the plane of the ester group is almost perpendicular to the naphthalene ring system, the angle being  $81.0(10)^\circ$ . The dihedral angle between the two naphthalene ring systems in (I) is  $105.3(10)^\circ$ . The large dihedral angle observed is not due to the presence of bulky bromine substituents. This can be concluded from the comparison of the structure of (I) with 1-naphthyl-2-phenylpropanoate (Gu *et al.* 2001), in which the planes of the aromatic rings are nearly orthogonal. The observed solid-state conformation of (I) precludes electron resonance/delocalization between ester and naphthyl groups. Furthermore, the puckered conformation of (I) is unsuitable for the photoinduced decarboxylation process due to the *anti* conformation of the  $\text{CH}_2-\text{O}-\text{CO}-\text{C}_{\text{aryl}}$  unit. The decarboxylation involves a concerted process which is favoured by the *syn* configuration of the unit.

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The crystal packing is dominated by van der Waals interactions.

## Experimental

A solution of 8-bromo-1-naphthoic acid (2.800 g) in thionyl chloride (30 ml) was refluxed for 10 h and concentrated under reduced pressure to yield 8-bromo-1-naphthoyl chloride as brown crystals. The chloride was dissolved in ethyl ether (20 ml) and added to a solution of lithium aluminium hydride (3.000 g) suspended in ethyl ether (20 ml). The mixture was refluxed for 10 h and cooled. Excess lithium aluminium hydride was removed by slow addition of a saturated sodium sulfate solution. After extracting with ethyl ether 3 times, the organic layer was dried with MgSO<sub>4</sub> and concentrated under reduced pressure, yielding a pale-yellow crude product 2.432 g which was chromatographed with a hexane–ethyl acetate (6:1) solution. Two main fractions were collected, the first fraction was yellow oil (0.150 g,  $R_F = 0.52$ ), the second fraction was the main fraction (2.001 g,  $R_F = 0.34$ ). Immediately after the chromatographic separation, colourless crystals appeared in the several tubes of the second fraction.

### Crystal data

$C_{22}H_{14}Br_2O_2$	$Z = 4$
$M_r = 470.15$	$D_x = 1.746 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 15.477 (7) \text{ \AA}$	$\mu = 4.55 \text{ mm}^{-1}$
$b = 8.824 (4) \text{ \AA}$	$T = 223 (2) \text{ K}$
$c = 14.006 (6) \text{ \AA}$	Plate, colourless
$\beta = 110.716 (8)^\circ$	$0.24 \times 0.24 \times 0.04 \text{ mm}$
$V = 1789.0 (13) \text{ \AA}^3$	

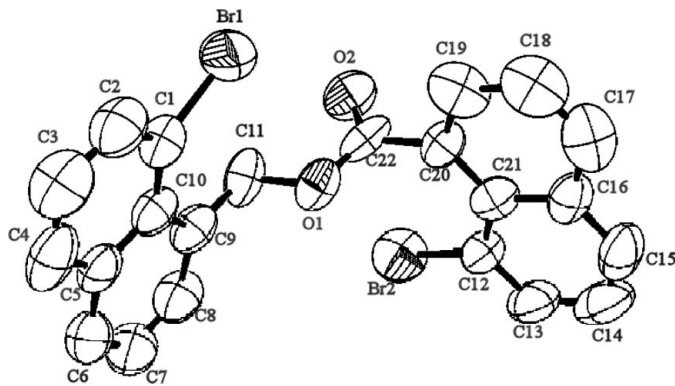
### Data collection

Bruker SMART CCD area-detector diffractometer	9904 measured reflections
$\varphi$ and $\omega$ scans	3153 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	1509 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.408$ , $T_{\max} = 0.839$	$R_{\text{int}} = 0.069$
	$\theta_{\max} = 25.0^\circ$

### Refinement

Refinement on $F^2$	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.071$	$w = 1/[\sigma^2(F_o^2) + (0.1218P)^2]$
$wR(F^2) = 0.236$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{\max} < 0.001$
3153 reflections	$\Delta\rho_{\max} = 0.64 \text{ e \AA}^{-3}$
235 parameters	$\Delta\rho_{\min} = -0.58 \text{ e \AA}^{-3}$

H atoms were positioned geometrically (C–H = 0.94 Å) and refined as riding, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .



**Figure 1**

The molecular structure of (I), showing 50% probability displacement ellipsoids. H atoms have been omitted.

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

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