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# 1-(3,4,5-Trimethoxyphenyl)naphthalene

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**Key indicators**

Single-crystal X-ray study
*T* = 296 K
Mean σ(C–C) = 0.004 Å
*R* factor = 0.038
*wR* factor = 0.098
Data-to-parameter ratio = 8.5

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

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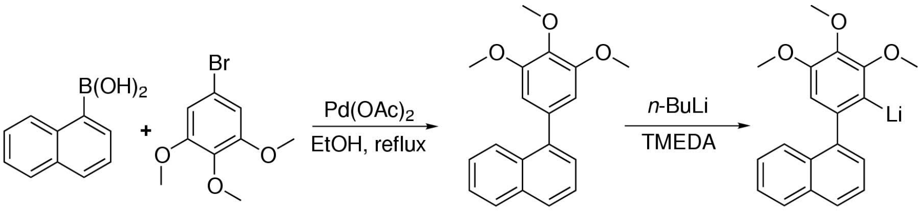
The title compound, $C_{19}H_{18}O_3$, is composed of a naphthalene ring, and a benzene ring with three substituents. The two H atoms on the benzene ring are enantiotopic; thus, one of them can be replaced with a bulky group to give an enantiomer of the resulting compound. The mean planes through the two ring systems are inclined to one another by 68.19 (10)°, with a central bridging bond of length 1.488 (3) Å.

## Comment

The title compound, (I), is of special interest because the two H atoms on the benzene ring with the three methoxy groups are enantiotopic; one of these can be replaced with a bulky group to give an enantiomer of the resulting compound. The X-ray study was carried out to verify that this structure was as anticipated and to determine structural details such as the torsion angle and the bond length between the two ring systems.



(I)

Compound (I) was synthesized using a modified Suzuki coupling (see scheme, first reaction) as described by Tao &

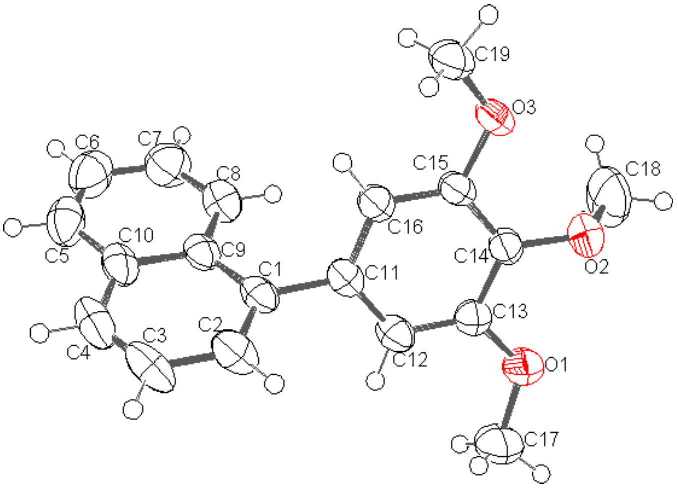


**Figure 1**
A view of the title compound, (I), showing 50% probability displacement ellipsoids.

Boykin (2004). It can be readily lithiated *ortho* to a methoxy group using *n*-BuLi and TMEDA in hexane at room temperature (see scheme). Further reaction of the lithiated compound with deuterium oxide yields the deuterated derivative. This derivative has been characterized by mass spectroscopy and $^1$H NMR. Mass spectroscopy demonstrated the expected increase of one mass unit, and $^1$H NMR integration verified that the lithium was indeed *ortho* to a methoxy group as expected.

The molecular structure of (I) is illustrated in Fig. 1. The mean planes through the two ring systems are inclined to one another by 68.19 (10)°. The C2—C1—C11—C12 torsion angle, between the two ring structures, is 66.1 (3)°, and the C1—C11 bond length is 1.488 (3) Å. This torsion angle is intermediate in value between the calculated *AM*1 and *PM*3 semi-empirical values of 58.5 and 71.6°, respectively.

Comparisons can be made with two published structures having similar ring systems, *viz*. 1-(*o*-tolyl)naphthalene (Boeyens *et al.*, 1988) and 1-(4,6-dimethoxy-2-methylphenyl)naphthalene (Peters *et al.*, 1995). Data in the Cambridge Structural Database (Version 5.26, ConQuest Version 1.7; Allen, 2002) were used to calculate the comparable torsion angle and bond length between the two ring systems. For the first structure (space group *Iba*2), these values were 97.2° and 1.488 Å, while for the second structure (space group $P2_1/n$), these values were 92.5° and 1.551 Å.

## Experimental

The title compound, (I), was synthesized using a modified Suzuki coupling as described by Tao & Boykin (2004). It was purified using automated flash chromatography, which yielded a viscous pale-yellow oil that solidified over the course of several weeks. Crystals suitable for X-ray diffraction were prepared by recrystallization from hot isooctane. $^1$H NMR (in CDCl$_3$): $\delta$ 3.88 (*s*, 6H), 3.95 (*s*, 3H), 6.71 (*d*, 2H), 7.4–7.55 (*m*, 4H), 7.86 (*d*, $J_{\rm HH}$ = 7.98 Hz, 1H), 7.91 (*d*, $J_{\rm HH}$ = 7.73 Hz, 1H), 7.96 (*d*, $J_{\rm HH}$ = 8.65 Hz, 1H); $^{13}$C NMR (in CDCl$_3$): $\delta$ 56.4 (CH$_3$), 61.2 (CH$_3$), 107.4 (CH), 125.5 (CH), 125.6 (CH), 126.2 (CH), 126.3 (CH), 126.9 (CH), 127.9 (CH), 128.5 (CH), 131.9 (C), 134.0 (C), 136.6 (C), 140.5 (C), 152.5 (C), 153.2 (CH); mass spectrum: $M^+$ 294 (100%), 279 (50%), 165 (52%).

### *Crystal data*

$C_{19}H_{18}O_3$
$M_r$ = 294.33
Orthorhombic, $P2_12_12_1$
$a$ = 6.746 (1) Å
$b$ = 12.605 (4) Å
$c$ = 18.320 (3) Å
$V$ = 1557.8 (6) Å$^3$
$Z$ = 4
$D_x$ = 1.25 Mg m$^{-3}$
Cu $K\alpha$ radiation
Cell parameters from 22 reflections
$\theta$ = 9.6–23.6°
$\mu$ = 0.68 mm$^{-1}$
$T$ = 296 (2) K
Prism, colourless
0.5 × 0.1 × 0.1 mm

### *Data collection*

Enraf–Nonius CAD-4 diffractometer
Non-profiled $\omega/2\theta$ scans
Absorption correction: none
3231 measured reflections
1722 independent reflections
1346 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.072
$\theta_{\rm max}$ = 69.9°
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 22$
3 standard reflections every 73 reflections intensity decay: 4%

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.098
$S$ = 1.05
1722 reflections
203 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 0.0081P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max}$ = 0.16 e Å$^{-3}$
$\Delta\rho_{\rm min}$ = −0.16 e Å$^{-3}$
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0022 (6)

The Flack (1983) parameter was refined to a value of 0.03 but with a standard uncertainty of 0.31. Friedel pairs were therefore merged and the $\Delta f''$ term set to zero before the final cycles of refinement. H atoms were constrained using a riding model. The aromatic C—H bond lengths were fixed at 0.93 Å, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C), and the methyl C—H bond lengths were fixed at 0.96 Å, with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C). For the methyl group, an idealized tetrahedral geometry was used, and the torsion angle about the bond to the methyl group was refined.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

The title compound, (I), was synthesized and the crystals prepared by Anthony Fowler and Dr Craig Ogle of the Department of Chemistry at the University of North Carolina at Charlotte. This work was supported in part by funds provided by The University of North Carolina at Charlotte.

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