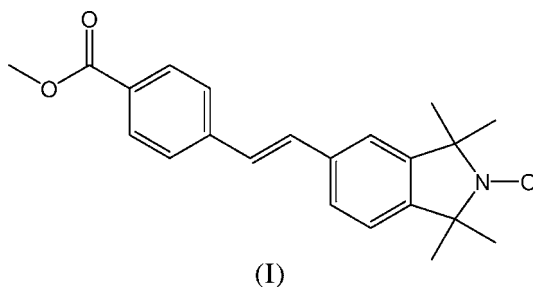# 5-[(E)-2-(4-Methoxycarbonylphenyl)ethenyl]-1,1,3,3-tetramethylisoindolin-2-yloxyl

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Key indicators

Single-crystal X-ray study
$T = 150$ K
Mean σ(C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.114
Data-to-parameter ratio = 19.2

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

The asymmetric unit of the title compound, $C_{22}H_{24}NO_3$, comprises two molecules with an E stereochemical configuration at the ethylene groups.

Comment

We are currently investigating the chemistry of paramagnetic profluorescent organic compounds. Of particular interest are the isoindoline class of nitroxide radicals (aminoxyls), such as TMIO (Smith *et al.*, 2000, 2002), as these display superior electron paramagnetic resonance (EPR) linewidths which results in increased accuracy in EPR oximetry (Bolton *et al.*, 1993; Gillies *et al.*, 1994; Bottle *et al.*, 1999, 2000; Shen *et al.*, 2002). Furthermore, the isoindoline system offers the opportunity to synthesize more complex structures through substitution of the aromatic ring, thus leading to the potential for advanced applications as radical sensors (Micallef *et al.*, 2005). The title compound, (I), was prepared as part of these ongoing investigations (Keddie *et al.*, 2005).

(I)

The asymmetric unit of (I), illustrated in Fig. 1, contains two molecules, both of which have an E (*trans*) stereochemical configuration at the ethylene bridge. The synthesis of this compound was performed by palladium-catalyzed Heck coupling of bromo-TMIO and methyl-4-vinylbenzoate. Both of these components are relatively bulky and were therefore expected to favour formation of the *trans* alkene product (Braese & Meijere, 2002). The nitroxide N—O bond lengths (Table 1) in the two molecules are typical for isoindoline nitroxide radicals (Busfield *et al.*, 1986; Fairhurst *et al.* 1996).

In the crystal structure of (I), the molecules are arranged in a zigzag-like pattern that propagates along the c axis. Interestingly, despite the obvious potential for intermolecular π–π interactions to occur between the benzene rings of the molecules of (I), there are no such interactions of significance apparent in this structure. The only directional interactions identified are methyl-C—H$\cdots\pi$ contacts (C21—H21$C\cdots$C3$^\mathrm{i}$–C8$^\mathrm{i}_\mathrm{plane}$ = 2.866 Å, C22—H22$C\cdots$C33–C38$_\mathrm{plane}$ = 2.688 Å, C43—H43$A\cdots$C25$^\mathrm{i}$–C30$^\mathrm{i}_\mathrm{plane}$ = 3.070 Å and C44—H44$C\cdots$C11$^\mathrm{ii}$–C16$^\mathrm{ii}_\mathrm{plane}$ = 2.852 Å).

organic papers

Experimental

The title compound was prepared by Heck coupling of 5-bromo-TMIO (Micallef *et al.*, 1999) and methyl-4-vinylbenzoate, according to the literature procedure reported by us (Keddie *et al.*, 2005). Slow evaporation of a 70:30 hexane–ethyl acetate solution of (I) afforded the large orange prismatic crystals used for this study.

Crystal data

$C_{22}H_{24}NO_3$	$Z = 8$
$M_r = 350.42$	$D_x = 1.222$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 11.6300$ (19) Å	$\mu = 0.08$ mm^{-1}
$b = 13.432$ (2) Å	$T = 150$ (2) K
$c = 24.407$ (4) Å	Prism, orange
$\beta = 91.976$ (3)°	$0.41 \times 0.37 \times 0.19$ mm
$V = 3810.5$ (11) Å^3	

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	37652 measured reflections
	9218 independent reflections
ω scans	7037 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1999)	$R_{int} = 0.030$
$T_{min} = 0.91$, $T_{max} = 0.99$	$\theta_{max} = 28.4°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0565P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.042$	$+ 0.9099P]$
$wR(F^2) = 0.114$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
9218 reflections	$\Delta\rho_{max} = 0.27$ e Å^{-3}
479 parameters	$\Delta\rho_{min} = -0.23$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected bond lengths (Å).

C9—C10	1.3328 (17)	N1—O3	1.2758 (13)
C31—C32	1.3297 (18)	N2—O6	1.2728 (14)

C-bound H atoms were included in idealized positions and refined using a riding model. Methine, aromatic and methyl C—H bond lengths were fixed at 0.95, 0.95 and 0.98 Å, respectively. U_{iso}(H) values were fixed at $1.2U_{eq}$(C) for methine and aromatic H atoms, and at $1.5U_{eq}$(C) for methyl H atoms.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT* and *XPREP* (Siemens, 1995); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *WinGX32* (Farrugia, 1999); software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004).
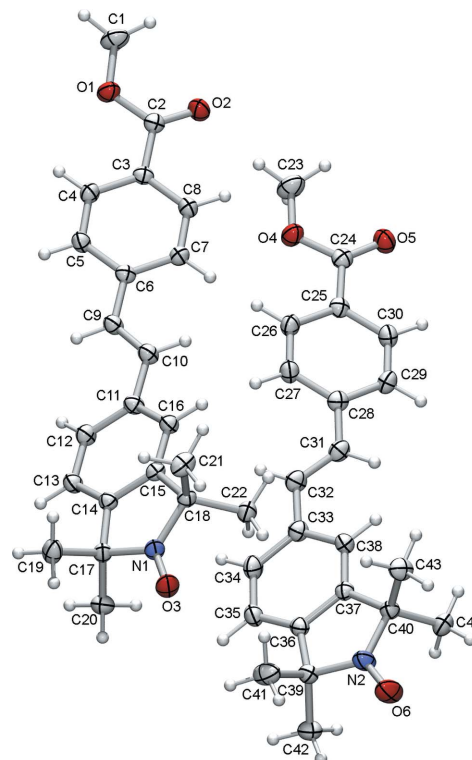
Figure 1
The two crystallographically independent molecules of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

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