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Methyl 4-(4-methoxybenzylidenemethyl)-3,5-dinitrobenzoate

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The title compound, $C_{17}H_{14}N_2O_7$, is a *trans*-stilbene and the dihedral angle between the benzene rings is 58.2 (1)°. The orientation of the nitro groups on the benzene ring is consistent with minimum steric interaction. In the solid state, inversion-related molecules are linked into dimers through C—H⋯O interactions and the translated dimers are linked *via* C—H⋯O interactions, thus forming a continuous chain running along one of the diagonals (011) of the *bc* plane.

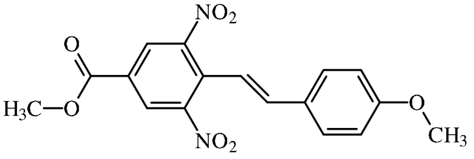
Key indicators

Single-crystal X-ray study
T = 293 K
Mean *σ*(C–C) = 0.002 Å
R factor = 0.046
wR factor = 0.145
Data-to-parameter ratio = 16.0

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

Comment

trans-Stilbene analogs are known as potent tyrosinase inhibitors and important constituents of depigmentation agents (Ohguchi *et al.*, 2003; Choi *et al.*, 2002). Recently, these derivatives have been shown to behave as apoptosis-inducing agents, based on striking inhibitory effects on associated cancer initiation (Roberti *et al.*, 2003; El-Zayat *et al.*, 1993). Stilbene systems are present in a number of medicinal plant compounds (Cuendet *et al.*, 2000; ElSohly *et al.*, 1984; Wanjala & Majinda, 2001) and are responsible for antioxidant (Biondi *et al.*, 2003; Fang *et al.*, 2002) and antimalarial activities (Boonlaksiri *et al.*, 2000). The X-ray crystal structure analysis of the title compound, 4′-methoxy-4-methoxycarbonyl-2,6-dinitrostilbene or methyl 4-(4-methoxybenzylidenemethyl)-3,5-dinitrobenzoate, (I), was carried out as part of our studies on stilbene derivatives.



(I)

In (I), the 4-methoxycarbonyl-2,6-dinitrophenyl ring is joined to the methoxyphenyl ring by a *trans*-ethene bridge. The N═O distances (Allen *et al.*, 1987; SethuSankar *et al.*, 2003) and Csp^2—N distances are comparable with earlier reported values (Gérard & Hardy, 1988). The length of the C7═C8 double bond is 1.315 (2) Å, which agrees with the reported values in the range 1.273 (11)–1.318 (3) Å (Hamazaki *et al.*, 1997; Allen *et al.*, 1987). The bond angle C7—C8—C9 [126.2 (1)°] is widened from 120° and this is due to the H7⋯H10 short contact of 2.172 Å.

The dihedral angle between the two benzene rings is 58.2 (1)°, which indicates that the *trans*-stilbene fragment is not planar. As expected, the nitro groups at C2 and C6 are twisted from the benzene ring and the dihedral angles are 60.3 (1) and 29.7 (1)°, respectively. These orientations are

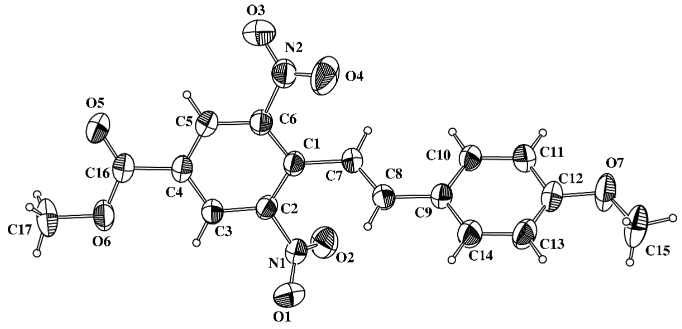


Figure 1
The molecular structure of the title compound, showing 35% probability displacement ellipsoids.

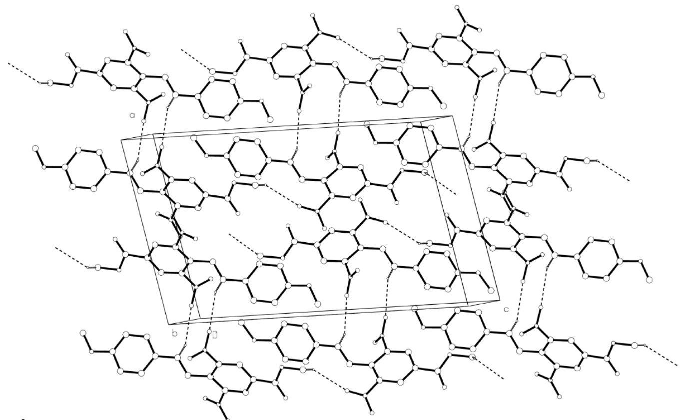


Figure 2
The molecular packing of (I), viewed down the *b* axis. Dashed lines indicate hydrogen bonds. For the sake of clarity, H atoms not involved in the hydrogen bonds have been omitted.

consistent with minimum steric interactions between the nitro groups and the ethene portion of the molecule. The methoxycarbonyl group is twisted out of the plane of the attached benzene ring [dihedral angle = 14.6 (1)° and C3—C4—C16—O6 = 14.7 (2)°]. The methoxy group is slightly twisted out of the plane of the attached benzene ring [C13—C12—O7—C15 = −5.5 (3)°].

The crystal packing is stabilized by C—H···O interactions (Table 2). Inversion-related molecules form a dimer, through a C8—H8···O1^{i} hydrogen bond, with graph-set descriptor $R_2^2(14)$ (Bernstein *et al.*, 1995). The translated dimers are linked through a C17—H17*A*···O4^{ii} hydrogen bond, forming a *C*(9) chain running along one of the diagonals (011) along the *bc* plane (Fig. 2) [symmetry codes: (i) $-x, -y, -z$; (ii) $x, \frac{1}{2}-y, z-\frac{1}{2}$].

Experimental

A mixture of 3,5-dinitro-4-methylbenzoic acid (5 mmol) and *p*-methoxybenzaldehyde (5 mmol) was stirred in dried dimethylformamide (25 mmol) in the presence of a catalytic amount of pyrrolidine. After stirring for 5 h, the reaction mixture was heated at 330 K for 30 min. It was then poured on to crushed ice and acidified with dilute HCl. The precipitate was filtered off, washed with methanol and dried over calcium chloride. Crystals of (I) were grown from acetone by slow evaporation.

Crystal data

$C_{17}H_{14}N_2O_7$
$M_r = 358.30$
Monoclinic, $P2_1/c$
$a = 12.0496 (8)$ Å
$b = 7.0295 (5)$ Å
$c = 19.8296 (13)$ Å
$\beta = 100.856 (1)°$
$V = 1649.56 (19)$ Å^3
$Z = 4$
$D_x = 1.443$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 2638 reflections
$\theta = 2.5–27.9°$
$\mu = 0.11$ mm^{-1}
$T = 293 (2)$ K
Block, pale yellow
0.26 × 0.20 × 0.12 mm

Data collection

Bruker SMART APEX diffractometer
ω scans
Absorption correction: none
9977 measured reflections
3785 independent reflections
2871 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.019$
$\theta_{\rm max} = 28.0°$
$h = -15 \rightarrow 7$
$k = -9 \rightarrow 9$
$l = -23 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.145$
$S = 1.02$
3785 reflections
237 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0837P)^2 + 0.21P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.28$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.22$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—N1	1.219 (2)	O4—N2	1.194 (2)
O2—N1	1.208 (2)	N1—C2	1.474 (2)
O3—N2	1.216 (2)	N2—C6	1.475 (2)
C2—C1—C7—C8	−50.5 (2)	C7—C8—C9—C10	−8.4 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O1^{i}	0.93	2.54	3.409 (2)	156
C17—H17*A*···O4^{ii}	0.96	2.54	3.139 (3)	120

Symmetry codes: (i) $-x, -y, -z$; (ii) $x, \frac{1}{2}-y, z-\frac{1}{2}$.

All H atoms were positioned geometrically and allowed to ride on their parent C atoms, with C—H distances in the range 0.93–0.96 Å and with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for methyl H atoms and $1.2U_{\rm eq}({\rm C})$ for the remainder. The methyl groups were allowed to rotate but not to tip.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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