Refinement

Refinement on F^2	$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$R(F)$ = 0.043	$\Delta\rho_{min}$ = −0.27 e Å^{-3}
$wR(F^2)$ = 0.073	Extinction correction:
S = 1.47	$I_o(\text{corr}) = I_o[1 + 2gI_c]$
1665 reflections	(Stout & Jensen, 1968)
166 parameters	Extinction coefficient:
All H atoms refined	g = 9.8 (13) × 10^{-7}
$w = 4F_o^2/[\sigma^2(I) + (0.02I)^2]$	Scattering factors from
$(\Delta/\sigma)_{max}$ = 0.001	Cromer & Waber (1974)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\sum_i\sum_j U^{ij}a^i a^j \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Cl	0.66388 (7)	3/4	0.55486 (8)	0.0569 (3)
Si	0.47333 (7)	3/4	0.5282	0.0335 (2)
C1	0.4193 (2)	0.6496 (1)	0.6002 (2)	0.0369 (5)
C2	0.2867 (2)	0.6262 (2)	0.5669 (2)	0.0497 (6)
C3	0.2552 (3)	0.5382 (2)	0.6117 (2)	0.0594 (8)
C4	0.2783 (3)	0.5278 (2)	0.7525 (3)	0.0597 (7)
C5	0.4077 (3)	0.5523 (1)	0.7886 (2)	0.0539 (7)
C6	0.4380 (2)	0.6403 (1)	0.7435 (2)	0.0434 (6)
C7	0.4462 (3)	3/4	0.3525 (3)	0.0351 (7)
C8	0.4939 (3)	0.8281 (1)	0.2851 (2)	0.0478 (7)
C9	0.4643 (3)	0.8277 (2)	0.1437 (2)	0.0571 (7)
C10	0.5127 (4)	3/4	0.0800 (3)	0.060 (1)

Table 2. *Selected geometric parameters (Å, °)*

Cl—Si	2.087 (1)	C3—C4	1.512 (3)
Si—C1	1.875 (2)	C4—C5	1.508 (4)
Si—C7	1.871 (3)	C5—C6	1.525 (3)
C1—C2	1.528 (3)	C7—C8	1.530 (2)
C1—C6	1.528 (3)	C8—C9	1.521 (3)
C2—C3	1.528 (3)	C9—C10	1.509 (3)
Cl—Si—C1	104.82 (7)	C3—C4—C5	111.9 (2)
Cl—Si—C7	106.78 (9)	C4—C5—C6	111.3 (2)
C1—Si—C1^i	118.5 (1)	C1—C6—C5	111.6 (2)
C1—Si—C7	110.47 (7)	Si—C7—C8	113.8 (1)
Si—C1—C2	114.4 (1)	C8—C7—C8^i	110.0 (2)
Si—C1—C6	116.2 (1)	C7—C8—C9	112.2 (2)
C2—C1—C6	109.1 (2)	C8—C9—C10	111.3 (2)
C1—C2—C3	111.6 (2)	C9—C10—C9^i	111.4 (3)
C2—C3—C4	111.5 (2)		

Symmetry code: (i) $x, \frac{3}{2} - y, z$.

Data were corrected for both decay and absorption. All non-H atoms were apparent in an E map. Refinement was on F^2; all reflections were used, with net negative intensities set at zero. All H atoms were located and refined isotropically. Refined C—H bond lengths were in the range 0.92 (2)–1.02 (2) Å; values of U_{iso}(H) were 0.035 (7)–0.074 (11) Å^2.

Data collection: *CAD-4/PC* (Enraf–Nonius, 1993). Cell refinement: *CAD-4/PC*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1989). Program(s) used to solve structure: *TEXSAN*. Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1098). Services for accessing these data are described at the back of the journal.

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(*E*)-1-(3-Methoxy-4-nitrophenyl)-2-(3,4,5-trimethoxyphenyl)ethene

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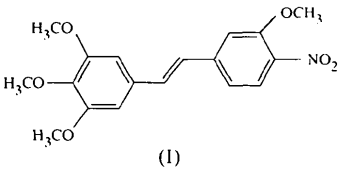
Abstract

The design and synthesis of new photoaffinity labeling (PAL) reagents is important in order to obtain detailed binding-site and structural information about tubulin. (*E*)-1-(3-Methoxy-4-nitrophenyl)-2-(3,4,5-trimethoxyphenyl)ethene, $C_{18}H_{19}NO_6$, crystallizes in the centrosymmetric space group $P2_1/n$ (No. 14), with four molecules in the unit cell. The *trans*-stilbene base component is nearly planar (mean deviation 0.05 Å), with a dihedral angle between the two phenyl rings of 6.1°. Important bond distances include C=C 1.326 (3) Å and mean N—O 1.187 (8) Å.

Comment

The design and synthesis of new photoaffinity labeling (PAL) reagents for tubulin is important in order to obtain detailed binding-site and structural information about this protein (Nare *et al.*, 1996; Sawada *et al.*, 1993; Rao *et al.*, 1992; Olszewski *et al.*, 1994). We have

prepared a number of potentially useful PAL reagents (Pinney & Rosenquist, 1995) which are structurally modeled around combretastatin A-4, a potent inhibitor of tubulin polymerization (Lin *et al.*, 1989; Pettit *et al.*, 1987). These ligands are designed to provide structural information concerning the colchicine binding site on tubulin (Pinney & Rosenquist, 1995). Although the *cis*-stilbenoid conformation in the combretastatin family is generally most advantageous for tubulin binding (Lin *et al.*, 1989; Pettit *et al.*, 1987; Cushman *et al.*, 1991), it is instructive to evaluate the binding affinity of certain *trans*-stilbenoid derivatives as well. In addition, it is important to correlate biological activity with structural details of the ligands. In order to expand the knowledge and research regarding tubulin, a detailed crystallographic analysis of (*E*)-1-(3-methoxy-4-nitrophenyl)-2-(3,4,5-trimethoxyphenyl)ethene, (I), is reported.



(I)

The title compound crystallizes in the centrosymmetric space group $P2_1/n$ (No. 14), with four molecules in the unit cell. The *trans*-stilbene molecule consists of a phenyl ring, with a nitro and a methoxy group, and a trimethoxyphenyl component joined by a *trans*-ethene bridge (Fig. 1). The bond lengths and angles (Table 1) are within expected ranges for this type of molecule and are in accord with published values found in BIDICS (1969–1981) and the Cambridge Structural Database (Orpen *et al.*, 1989). The mean N—O, C_{sp^3}—O and C_{sp^2}—O distances are 1.187 (8), 1.415 (8) and 1.37 (1) Å, respectively.

The two phenyl rings are planar, with mean deviations from the best least-squares plane of 0.006 Å for the nitromethoxyphenyl ring and 0.013 Å for the trimethoxyphenyl ring. The nitro group is slightly twisted out of the plane of the attached phenyl ring (dihedral angle 5.3° and C11—C12—N—O5 4.0°). The dihedral angle between the two phenyl components is 6.1°, which indicates that the *trans*-stilbene base complex is nearly planar (mean deviation from the best least-squares plane is 0.06 Å). The C1—C7—C8—C9 torsion angle of −178.4 (2)° provides further evidence that the stilbene component is planar.

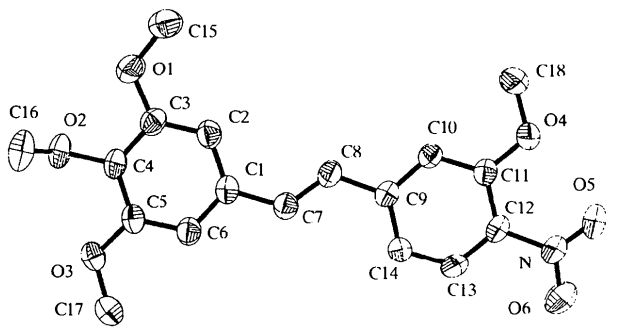


Fig. 1. The molecular structure of the title compound showing the atom-labeling scheme and displacement ellipsoids at the 50% probability level. H atoms have been omitted for clarity.

Experimental

A solution of (3,4,5-trimethoxybenzyl)triphenylphosphonium bromide (1.50 g, 2.86 mmol) and 3-methoxy-4-nitrobenzaldehyde (0.518 g, 2.86 mmol) in CH_2Cl_2 (50 ml) was stirred under a nitrogen atmosphere. After 30 min, NaH (0.412 g, 17.16 mmol) was added. After 16 h, water was added and the product was isolated by extraction with CH_2Cl_2. The organic phase was washed with brine and dried over $MgSO_4$. Purification by flash chromatography (silica gel, 70:30 hexanes–ethyl acetate) afforded the ethene (0.274 g, 0.793 mmol, 28%) as a bright yellow solid. Recrystallization (hexanes–CH_2Cl_2) afforded an analytically pure sample of the title compound.

Crystal data

$C_{18}H_{19}NO_6$
M_r = 345.3
Monoclinic
$P2_1/n$
a = 11.027 (3) Å
b = 12.1065 (12) Å
c = 13.199 (3) Å
β = 106.45 (2)°
V = 1689.9 (6) Å^3
Z = 4
D_x = 1.36 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 3.87–12.00°
μ = 0.096 mm^{-1}
T = 292 K
Irregular parallelepiped
0.52 × 0.39 × 0.25 mm
Clear yellow

Data collection

Enraf–Nonius CAD-4*F* diffractometer
ω–2θ scans
Absorption correction: none
3299 measured reflections
2967 independent reflections
2234 reflections with $F > 4\sigma(F)$

R_{int} = 0.013
θ_{max} = 25°
h = −13 → 13
k = 0 → 14
l = 0 → 15
3 standard reflections
frequency: 120.0 min
intensity decay: 0.2%

Refinement

Refinement on F
R = 0.045
wR = 0.071
S = 1.805
2234 reflections
227 parameters
H atoms constrained
$w = 1/[\sigma^2(F) + 0.002F^2]$
$(\Delta/\sigma)_{max}$ = 0.002

$\Delta\rho_{max}$ = 0.286 e Å^{-3}
$\Delta\rho_{min}$ = −0.241 e Å^{-3}
Extinction correction: *SHELXTL-Plus*
Extinction coefficient: 0.0010 (5)
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

C1—C2	1.398 (3)	C9—C14	1.403 (3)
C1—C6	1.395 (3)	C10—C11	1.388 (3)
C1—C7	1.464 (3)	C11—C12	1.407 (3)
C2—C3	1.386 (3)	C11—O4	1.354 (3)
C3—C4	1.393 (3)	C12—C13	1.393 (3)
C3—O1	1.364 (2)	C12—N	1.446 (3)
C4—C5	1.392 (3)	C13—C14	1.363 (3)
C4—O2	1.383 (2)	O1—C15	1.405 (3)
C5—C6	1.387 (3)	O2—C16	1.412 (4)
C5—O3	1.368 (3)	O3—C17	1.417 (3)
C7—C8	1.326 (3)	O4—C18	1.427 (3)
C8—C9	1.470 (3)	N—O5	1.179 (3)
C9—C10	1.390 (3)	N—O6	1.194 (3)
C2—C1—C7	123.5 (2)	C10—C11—O4	123.5 (2)
C6—C1—C7	117.2 (2)	C12—C11—O4	118.5 (2)
C2—C3—O1	125.0 (2)	C11—C12—N	122.7 (2)
C4—C3—O1	114.9 (2)	C13—C12—N	117.2 (2)
C3—C4—O2	120.1 (2)	C3—O1—C15	117.9 (2)
C5—C4—O2	120.0 (2)	C4—O2—C16	113.7 (2)
C4—C5—O3	115.7 (2)	C5—O3—C17	117.8 (2)
C6—C5—O3	124.3 (2)	C11—O4—C18	118.1 (2)
C1—C7—C8	128.1 (2)	C12—N—O5	121.8 (2)
C7—C8—C9	124.6 (2)	C12—N—O6	118.6 (2)
C8—C9—C10	118.9 (2)	O5—N—O6	119.6 (2)
C8—C9—C14	122.2 (2)		
C2—C1—C7—C8	−11.4 (4)	C7—C8—C9—C14	5.8 (4)
C2—C3—O1—C15	−8.4 (3)	C10—C11—O4—C18	−13.1 (3)
C3—C4—O2—C16	−90.3 (3)	C11—C12—N—O5	4.0 (4)
C6—C5—O3—C17	2.1 (4)	C13—C12—N—O6	3.1 (4)
C1—C7—C8—C9	−178.4 (2)		

A conoscopic examination between two crossed polarizers on a Zeiss Photomicroscope II confirmed the optical quality and nature of the crystal. The structure was checked for additional symmetry with the *MISSYM* program (Gabe *et al.*, 1989).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *SDP* (Enraf–Nonius, 1985). Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1989) (direct methods). Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *CIFGEN* (local program).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SX1050). Services for accessing these data are described at the back of the journal.

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