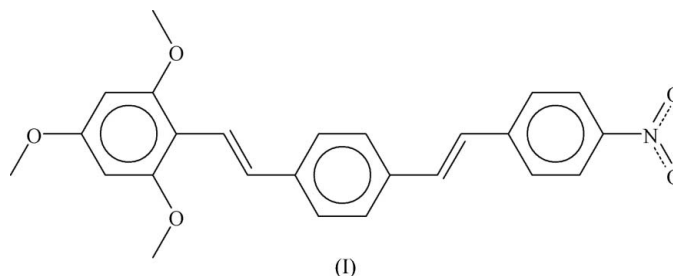
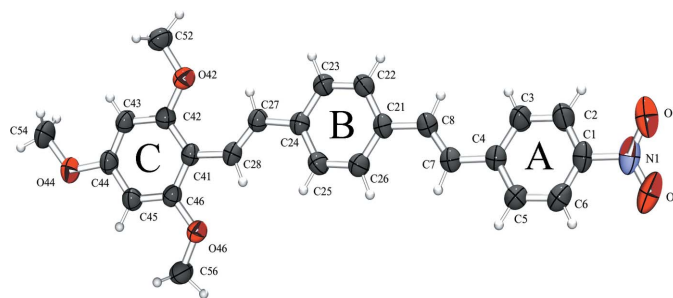


(*E,E*)-1-[2-(4-Nitrophenyl)ethenyl]-4-[2-(2,4,6-trimethoxyphenyl)ethenyl]benzene**Christophe M. L. Vande Velde,
Roeland De Borger and Frank
Blockhuys***Department of Chemistry, University of
Antwerp, Universiteitsplein 1, B-2610 Wilrijk,
BelgiumCorrespondence e-mail:
frank.blockhuys@ua.ac.be**Key indicators**Single-crystal X-ray study
 $T = 287$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.040
 wR factor = 0.121
Data-to-parameter ratio = 10.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The crystal structure of the title compound, $\text{C}_{25}\text{H}_{23}\text{NO}_5$, is centrosymmetric; it crystallizes in ribbons interacting through $\text{C}-\text{H} \cdots \pi$ contacts and $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds.Received 27 October 2005
Accepted 21 November 2005
Online 26 November 2005**Comment**The title compound, (I) (Fig. 1), was synthesized as a material for organic memories (Bandyopadhyay & Pal, 2003). Since its properties as a memory material are closely related to its crystal structure, a determination of the latter is mandatory. Compound (I) is only the second asymmetric distyrylbenzene so far for which the crystal structure has been reported, the first being (*E,E*)-2,5-difluoro-1-(pentafluorostyryl)-4-(4-*tert*-butylstyryl)benzene [CSD (Cambridge Structural Database; Version 5.26, update of August 2005; Allen, 2002) refcode REFKUI (Bartholomew *et al.*, 2000)]. The present paper deals with the structural features of (I).The molecular structure contains no surprises in either bond lengths or angles. It is, however, striking that the central benzene ring *B* displays considerable quinoid character, as can be seen from the selected geometric parameters in Table 1. In REFKUI, the same phenomenon can be observed.The vinylic spacers around the central ring have a *syn* conformation, meaning that the divinylbenzene part of the**Figure 1**
View of (I), including the numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

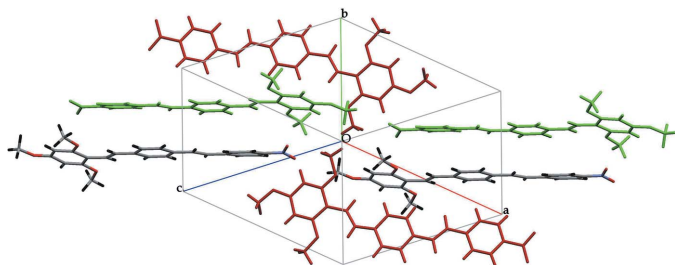


Figure 2

View of the structure along a cell body diagonal. The original molecule $[(x, y, z)]$, on the left and that related through translation symmetry along the a and negative c axes $(x + 1, y, z - 1)$ are drawn in green. The symmetry equivalents of these through the inversion centre are drawn in conventional colours $[(-x, 1 - y, 2 - z)]$ and $(1 - x, 1 - y, 1 - z)]$ and those through the glide planes are drawn in maroon $[(x, \frac{3}{2} - y, z - \frac{1}{2})]$ and $(1 - x, y - \frac{1}{2}, \frac{3}{2} - z)]$.

molecule has approximate C_{2h} symmetry as opposed to the C_i symmetry that is normally found in the structures of (*E,E*)-distyrylbenzene derivatives. In the CSD, only one other distyrylbenzene shows such a *syn* arrangement, namely 4,4'-bis(perfluorostyryl)stilbene (refcode QERWUF; Feast *et al.*, 2001). The angle between ring *A* and ring *B* is $8.3 (1)^\circ$, and the angle between ring *B* and ring *C* is $11.0 (1)^\circ$; the angle between ring *A* and ring *C* is $17.9 (1)^\circ$.

The molecule crystallizes in the centrosymmetric space group $P2_1/c$, yielding a crystal structure in which the dipoles are lined up between cofacially stacked pairs of molecules related through translation/inversion symmetry. These pairs of molecules form extended double ribbons along the $[\bar{1}01]$ direction, as shown in Fig. 2. The symmetry-equivalent molecules through the glide planes form their own ribbons in the same $[\bar{1}01]$ direction.

The mutually perpendicular ribbons interact through typical herringbone $C-H \cdots \pi$ contacts originating from the nitro-substituted ring *A* to rings *B* and *C*. These contacts are designated by $C5-H5 \cdots Cg(B)^i$ [symmetry code: (i) $x, \frac{1}{2} - y, \frac{1}{2} + z$; $H5 \cdots Cg(B)^i = 2.71 (2) \text{ \AA}$, 2.646 \AA perp., $C5-H5 \cdots Cg(B)^i = 167.0 (19)^\circ$] and $C3-H3 \cdots Cg(C)^{ii}$ [symmetry code: (ii) $-x, -\frac{1}{2} + y, \frac{1}{2} - z$; $H3 \cdots Cg(C)^{ii} = 2.96 (2) \text{ \AA}$, 2.746 \AA perp., $C3-H3 \cdots Cg(C)^{ii} = 140.7 (18)^\circ$], where $Cg(X)$ denotes the centroid of ring *X* and 'perp.' indicates the perpendicular distance of the H atom from the plane of the ring. Between these same molecules, H2 displays the contacts $H2 \cdots O44^{ii} = 2.65 (2) \text{ \AA}$, $C2-H2 \cdots O44 = 155 (2)^\circ$ and $H2 \cdots C44^{ii} = 2.89 (2) \text{ \AA}$, $C2-H2 \cdots C44 = 127 (2)^\circ$. The other short contacts present in the structure are weak hydrogen bonds involving the O atoms of the nitro group: $C52-H521 \cdots O(2)^{iii}$ [symmetry code: (iii) $-x, -y, 1 - z$; $2.49 (2) \text{ \AA}$, $136.5 (18)^\circ$], $C54-H543 \cdots O(2)^{iv}$ [symmetry code: (iv) $1 + x, y, -1 + z$; $2.71 (3) \text{ \AA}$, $122 (2)^\circ$] and $C45-H45 \cdots O1^v$ [symmetry code: (v) $1 + x, \frac{1}{2} - y, -\frac{1}{2} + z$; $2.32 (2) \text{ \AA}$, $158.0 (18)^\circ$]. A final interaction, which could possibly account for the twist of the nitro group out of the plane of the molecule, is $N1-O1 \cdots Cg(C)^{iii}$ [$3.267 (3) \text{ \AA}$, 3.195 \AA perp., $106.65 (16)^\circ$].

A TLS analysis with *PLATON* (Spek, 2003) reveals that the main librational component L1 lies along the long axis of the molecule and is larger than the other two (7.75° versus 1.78°

and 1.23°), but not by a whole order of magnitude as is usual for these compounds. The pair-wise ordering of the molecules by the action of the crystallographic centre of symmetry appears to hinder the librational motion along the long axis of the molecule to a large extent.

Experimental

Compound (I) was synthesized starting from (*E*)-1-(4-methylphenyl)-2-(4-nitrophenyl)ethene (II) (De Borger *et al.*, 2005). For the synthesis of (*E*)-1-(4-bromomethylphenyl)-2-(4-nitrophenyl)ethene, (III), *N*-bromosuccinimide (0.04 mol) and a catalytic amount of 1,1'-azobis(cyclohexanecarbonitrile) were added to a heated solution of (II) (0.032 mol) in CCl_4 (150 ml). The mixture was refluxed overnight. The hot solution was filtered to remove the succinimide. The solvent was then partially evaporated and the precipitated yellow compound was collected by filtration in a yield of 63% [m.p. 383 K (uncorrected)]. (*E*)-Triphenyl[4-(*p*-nitrostyryl)benzyl]phosphonium bromide, (IV), was prepared by adding triphenylphosphine (0.025 mol) to a solution of (III) (0.023 mol) in acetonitrile (150 ml). The mixture was refluxed for 4 h. After cooling to room temperature, the product was collected by filtration, washed with diethyl ether and used without further characterization. The yield was 72% [m.p. 563 K (decomposition)]. Finally, (I) was prepared by dropwise addition of sodium (0.0165 mol) in dry ethanol (40 ml) to a stirred mixture of (IV) (0.0165 mol) and 2,4,6-trimethoxybenzaldehyde (0.0165 mol) in the same solvent (100 ml). The mixture was refluxed in a nitrogen atmosphere for 6 h. After cooling to room temperature, water (125 ml) was added. The mixture was filtered and the compound washed with diethyl ether. To obtain the (*E,E*) isomer, the compound was refluxed in *p*-xylene containing a catalytic amount of iodine for 4 h. After cooling, the product crystallized as X-ray quality crystals, which were collected by filtration. The yield was 44% [m.p. 451 K (uncorrected)]. 1H NMR ($CDCl_3$, 400 MHz, TMS): δ 3.85 (s, 3H, H54), 3.90 (s, 6H, H52 and H56), 6.18 (s, 2H, H43 and H45), 7.12 (d, 1H, $J = 16.33$ Hz, H7), 7.26 (d, 1H, $J = 16.33$ Hz, H8), 7.47 (s, 2H, H27 and H28), 7.50 (d, 2H, $J = 8.69$, H23 and H25), 7.53 (d, 2H, $J = 8.70$, H22 and H26), 7.62 (d, 2H, $J = 8.85$ Hz, H3 and H5), 8.21 (d, 2H, $J = 8.85$ Hz, H2 and H6). ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 55.4 (C54), 55.8 (C52 and C56), 91.0 (C43 and C45), 108.2 (C41), 120.8 (C27), 124.1 (C2 and C6), 125.2 (C7), 126.6 (C3 and C5), 126.7 (C23 and C25), 127.3 (C22 and C26), 129.1 (C8), 133.4 (C28), 134.3 (C24), 140.7 (C21), 144.2 (C4), 146.6 (C1), 159.7 (C42 and C46), 160.6 (C44).

Crystal data

$C_{25}H_{23}NO_5$	$D_x = 1.306 \text{ Mg m}^{-3}$
$M_r = 417.44$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 25 reflections
$a = 15.785 (5) \text{ \AA}$	$\theta = 7.5\text{--}21.8^\circ$
$b = 10.357 (2) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$c = 13.502 (1) \text{ \AA}$	$T = 287 (2) \text{ K}$
$\beta = 105.96 (2)^\circ$	Rhomboïd, red–orange
$V = 2122.2 (8) \text{ \AA}^3$	$0.3 \times 0.2 \times 0.2 \text{ mm}$
$Z = 4$	

Data collection

Enraf–Nonius MACH3 diffractometer	$\theta_{\max} = 25.3^\circ$
$\omega/2\theta$ scans	$h = -18 \rightarrow 18$
Absorption correction: none	$k = -12 \rightarrow 4$
4890 measured reflections	$l = -16 \rightarrow 0$
3880 independent reflections	3 standard reflections
2320 reflections with $I > 2\sigma(I)$	frequency: 60 min
$R_{\text{int}} = 0.011$	intensity decay: 1.5%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.121$
 $S = 1.03$
 3880 reflections
 372 parameters
 All H-atom parameters refined

$$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.3937P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.18 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.17 \text{ e } \text{\AA}^{-3}$

Table 1

Selected bond lengths (Å).

C4—C7	1.464 (3)	C24—C27	1.458 (3)
C7—C8	1.324 (3)	C27—C28	1.328 (3)
C8—C21	1.460 (3)	C28—C41	1.460 (3)
C21—C22	1.393 (3)	C24—C25	1.399 (3)
C22—C23	1.374 (3)	C21—C26	1.395 (3)
C23—C24	1.396 (3)	C26—C25	1.378 (3)

All H atoms were located in a difference density map and refined freely [C—H = 0.90 (2)–1.04 (3) Å].

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *DREAR* (Blessing, 1987); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *MERCURY* (Bruno *et al.*, 2002) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare

material for publication: *PLATON* (Spek, 2003) and *WinGX* (Farrugia, 1999).

CVV thanks the FWO Vlaanderen for a grant as a research assistant. The authors thank Professor Dr R. Dommissie and J. Aerts for the NMR measurements.

References

- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
- Bandyopadhyay, A. & Pal, A. J. (2003). *Appl. Phys. Lett.* **82**, 1215–1217.
- Bartholomew, G. P., Bazan, G. C., Bu, X. & Lachicotte, R. J. (2000). *Chem. Mater.* **12**, 1422–1430.
- Blessing, R. H. (1987). *Crystallogr. Rev.* **1**, 3–58.
- Bruno, I. J., Cole, J. C., Edgington, P. R., Kessler, M., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). *Acta Cryst.* **B58**, 389–397.
- De Borger, R., Vande Velde, C. M. L. & Blockhuys, F. (2005). *Acta Cryst.* **E61**, o819–o821.
- Enraf–Nonius (1994). *CAD-4 EXPRESS*. Enraf–Nonius, Delft, The Netherlands.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Feast, W. J., Lövenich, P. W., Puschmann, H. & Taliani, C. (2001). *Chem. Commun.* pp. 505–506.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.