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trans-Ethylenedi-*p*-phenylene diacetate

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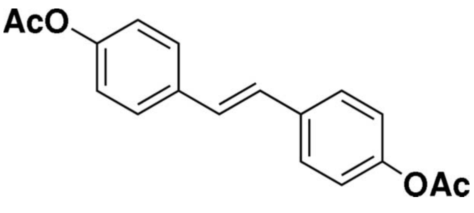
Key indicators: single-crystal X-ray study; *T* = 100 K; mean σ(C–C) = 0.002 Å; *R* factor = 0.042; *wR* factor = 0.106; data-to-parameter ratio = 15.8.

The centrosymmetric title compound, $C_{18}H_{26}O_4$, was prepared in high yield from 4-acetoxystyrene *via* Ru-catalysed homo-olefin metathesis. Exclusive formation of the *E*-configurated isomer was observed. In the crystal, a strong C—H$\cdots\pi$ intermolecular interaction links the molecules together.

Related literature

For the preparation of differently substituted stilbenes using a Ru-catalysed metathesis strategy, see: Velder *et al.* (2006). For alternative methodologies for the synthesis of oxy-functionalized stilbenes using Wittig-type olefinations or Heck-couplings, see: Kim *et al.* (2002); Lion *et al.* (2005); Botella *et al.* (2004); Reetz *et al.* (1998). For the bioactivity of various stilbenes with a focus on their anticancer activity, see: Aggarwal *et al.* (2004); Wolter *et al.* (2002); Fremont (2000); Jang *et al.* (1997); Wieder *et al.* (2001). For related structures see: Malone *et al.* (1997). For a previous synthesis of the title compound see: Johnson *et al.* (1952).



Experimental

Crystal data

$C_{18}H_{16}O_4$
M_r = 296.31
Monoclinic, $P2_1/c$
a = 9.7430 (4) Å
b = 7.2839 (4) Å
c = 11.2723 (6) Å
β = 113.649 (3)°
V = 732.78 (7) $Å^3$
Z = 2
Mo *K*α radiation
μ = 0.10 mm^{-1}
T = 100 K
0.52 × 0.36 × 0.34 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
3533 measured reflections
1595 independent reflections
1119 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.106
S = 1.03
1595 reflections
101 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.21 e $Å^{-3}$

Table 1
Geometry the C—H$\cdots\pi$ interaction (Å, °).

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
C7—H7$\cdots$*Cg*1^{i}	0.95	2.81	3.539 (2)	135

Symmetry code: (i) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$. *Cg*1 is the centroid of the C2–C7 ring.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SCHAKAL99* (Keller, 1999); software used to prepare material for publication: *PLATON* (Spek, 2009) and *enCIFer* (Allen *et al.*, 2004).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2554).

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trans-Ethylenedi-*p*-phenylene diacetate

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S1. Comment

Resveratrol-related stilbenes exhibit promising anticancer activity (Aggarwal *et al.*, 2004; Wolter *et al.*, 2002; Fremont *et al.*, 2000; Jang *et al.*, 1997). Based on our own research in the field of bioactive stilbenes (Wieder *et al.*, 2001) we decided to reinvestigate the possibility of using a cross-metathesis strategy for the synthesis of compounds of type 1 (Velder *et al.*, 2006) which turned out to be a highly efficient route towards symmetrically as well as unsymmetrically substituted E-stilbenes. Alternative strategies for the synthesis of stilbenes are based on Wittig-type olefinations or Heck couplings (Kim *et al.* (2002), Lion *et al.* (2005), Botella *et al.* (2004), Reetz *et al.* (1998)). One of the compounds prepared is the title compound *trans*-1,2-bis-(4-acetoxyphenyl)ethene. Within each molecule the two planes defined by the arene moieties are co-planar but slightly stepped (by 0.324 (2) Å) due to the fact that the plane defined by the central double bond is twisted by a torsion angle of -13.8 (2)° (C1a—C1—C2—C7) and 165.7 (15)° (C1a—C1—C2—C3), respectively (figure 1). The molecules form layers which are intermolecularly linked through a C—H···π interaction of type III (Malone *et al.* 1997). This interaction occurs between the H atom of one phenyl group and the π-system of the other phenyl moiety (figure 2). With a H···π distance of only 2.77 Å these interactions are rather strong.

S2. Experimental

In a glove-box (Labmaster 130, mBraun), the catalyst (Grubbs-II, 2 mol %) was weighted into a 25 ml Schlenk tube, which was sealed with a rubber septum. This was then taken out of the box, connected to an Ar-vacuum double manifold and equipped with a reflux condenser under argon. A solution of 3-acetoxy-styrene (1.0 g, 6.17 mmol) in CH_2Cl_2 (20 ml) was added *via* syringe and the resulting solution was refluxed for 1.5 h under argon. After allowing the reaction mixture to cool to room temperature, the solvent was evaporated *in vacuo* and the crude product was purified by recrystallization from EtOAc/cyclohexane 5:1 to give 0.8 g (88%) of the homo-metathesis product 1. mp. 214 °C (Johnson *et al.* (1952) 215–218°C). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.29 (s, 3H, CH_3), 7.04 (s, 1H, CH=), 7.08 (d, 2H, J = 8.7 Hz, H-3, H-5), 7.49 (d, 2H, J = 8.7 Hz, H-2, H-6); ^{13}C NMR (300 MHz, $CDCl_3$): δ = 21.2 (CH_3), 121.8 (C-3, C-5), 127.4 (C-2, C-6), 127.9 (C-7), 135.0 (C-1), 150.1 (C-4), 169.5 (C=O); HRMS, calcd for $C_{18}H_{16}O_4$ (M^+) 296.1048, found 296.105.

S3. Refinement

Hydrogen atoms were located in difference syntheses, and are refined at idealized positions (C—H = 0.98Å for methyl H atoms and 0.95Å for all other H Atoms) using a riding model, the U values of the H atoms are constrained relative to U_{eq} of the parent carbon atom (1.2 *x* U_{eq}(C) for C—H and 1.5 *x* U_{eq}(C) for methyl H).

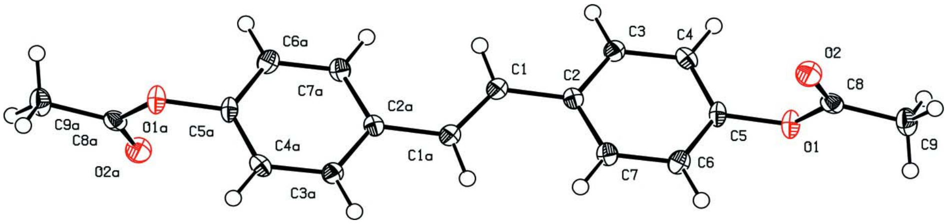

Figure 1

A top view of 1. Displacement ellipsoids are drawn at the 50% probability level.

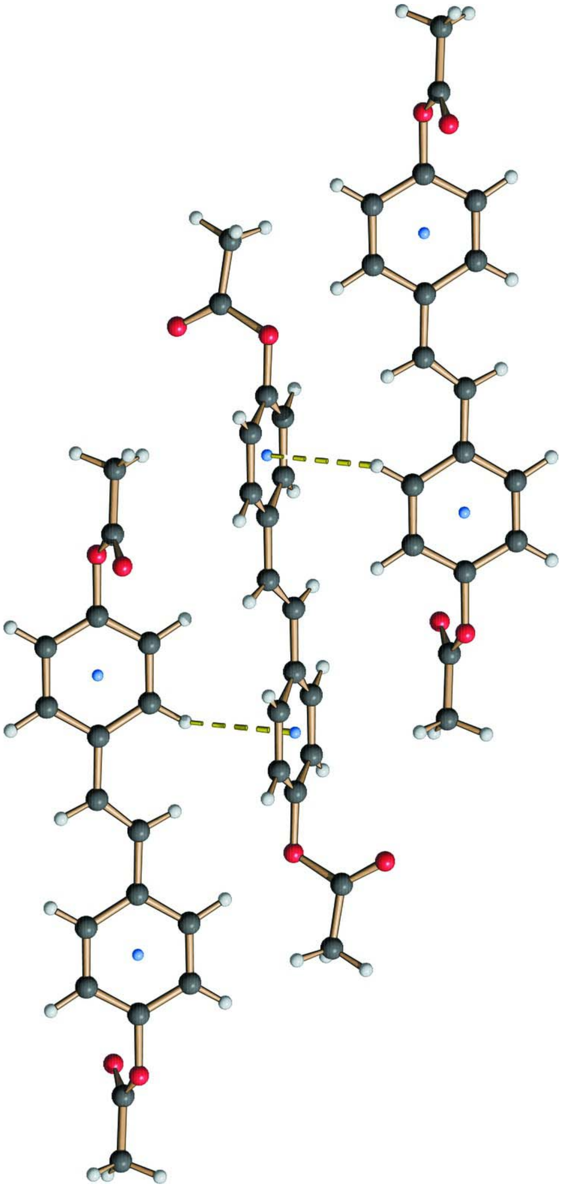

Figure 2

Intramolecular C—H···π interactions.

***trans*-Ethylenedi-*p*-phenylene diacetate**

Crystal data

$C_{18}H_{16}O_4$
$M_r = 296.31$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.7430$ (4) Å
$b = 7.2839$ (4) Å
$c = 11.2723$ (6) Å
$\beta = 113.649$ (3)°
$V = 732.78$ (7) Å^3
$Z = 2$

$F(000) = 312$
$D_x = 1.343$ Mg m^{-3}
Melting point: 214 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3533 reflections
$\theta = 2.3$–27.0°
$\mu = 0.10$ mm^{-1}
$T = 100$ K
Needle, colourless
0.52 × 0.36 × 0.34 mm

Data collection

Nonius KappaCCD
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
3533 measured reflections
1595 independent reflections

1119 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.038$
$\theta_{\rm max} = 27.0°$, $\theta_{\rm min} = 2.3°$
$h = -12 \rightarrow 12$
$k = -8 \rightarrow 9$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.106$
$S = 1.03$
1595 reflections
101 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.0561P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.002$
$\Delta\rho_{\rm max} = 0.20$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.21$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger. The coordinates of the hydrogen atoms are constrained, and the U values of the H atoms are constrained relative to the $U_{\rm eq}$ of the atom the hydrogen binds to (1.2 for CH and CH_2, 1.5 for CH_3).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
O1	0.26496 (12)	0.47976 (14)	0.58904 (9)	0.0210 (3)
O2	0.37476 (12)	0.75603 (15)	0.60504 (10)	0.0249 (3)
C1	0.06877 (16)	0.48067 (19)	0.04361 (14)	0.0165 (3)

H1	0.1412	0.4466	0.0111	0.020*
C2	0.11855 (16)	0.48580 (19)	0.18473 (14)	0.0151 (3)
C3	0.25433 (16)	0.4033 (2)	0.26319 (14)	0.0171 (3)
H3	0.3134	0.3468	0.2238	0.020*
C4	0.30488 (16)	0.4019 (2)	0.39715 (14)	0.0176 (3)
H4	0.3971	0.3446	0.4492	0.021*
C5	0.21842 (17)	0.4853 (2)	0.45310 (13)	0.0165 (4)
C6	0.08410 (17)	0.5691 (2)	0.37923 (14)	0.0189 (4)
H6	0.0261	0.6259	0.4195	0.023*
C7	0.03502 (17)	0.5694 (2)	0.24605 (14)	0.0179 (4)
H7	−0.0572	0.6274	0.1950	0.021*
C8	0.34023 (17)	0.6307 (2)	0.65644 (15)	0.0187 (4)
C9	0.37163 (18)	0.6128 (2)	0.79670 (14)	0.0245 (4)
H9A	0.4210	0.7245	0.8424	0.037*
H9B	0.4373	0.5070	0.8330	0.037*
H9C	0.2773	0.5951	0.8069	0.037*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0304 (7)	0.0177 (6)	0.0123 (6)	−0.0031 (5)	0.0058 (5)	−0.0012 (4)
O2	0.0264 (7)	0.0227 (6)	0.0258 (6)	−0.0058 (5)	0.0106 (5)	−0.0025 (5)
C1	0.0191 (8)	0.0139 (8)	0.0174 (8)	−0.0008 (6)	0.0083 (6)	−0.0011 (6)
C2	0.0168 (8)	0.0117 (7)	0.0154 (8)	−0.0031 (6)	0.0051 (6)	0.0008 (6)
C3	0.0184 (8)	0.0156 (8)	0.0175 (8)	−0.0012 (6)	0.0076 (7)	−0.0019 (6)
C4	0.0156 (8)	0.0154 (8)	0.0181 (8)	−0.0004 (6)	0.0027 (6)	0.0013 (6)
C5	0.0239 (9)	0.0132 (8)	0.0106 (8)	−0.0049 (6)	0.0049 (7)	−0.0005 (6)
C6	0.0245 (9)	0.0145 (8)	0.0192 (8)	0.0008 (6)	0.0102 (7)	−0.0023 (6)
C7	0.0198 (9)	0.0152 (8)	0.0169 (8)	0.0018 (6)	0.0055 (7)	0.0010 (6)
C8	0.0146 (8)	0.0187 (9)	0.0212 (8)	0.0035 (6)	0.0056 (7)	−0.0039 (7)
C9	0.0275 (9)	0.0245 (9)	0.0174 (9)	0.0029 (7)	0.0046 (7)	−0.0037 (7)

Geometric parameters (Å, º)

O1—C8	1.3700 (18)	C4—C5	1.380 (2)
O1—C5	1.4135 (16)	C4—H4	0.9500
O2—C8	1.1994 (17)	C5—C6	1.380 (2)
C1—C1$^{\mathrm{i}}$	1.336 (3)	C6—C7	1.381 (2)
C1—C2	1.466 (2)	C6—H6	0.9500
C1—H1	0.9500	C7—H7	0.9500
C2—C3	1.398 (2)	C8—C9	1.491 (2)
C2—C7	1.401 (2)	C9—H9A	0.9800
C3—C4	1.388 (2)	C9—H9B	0.9800
C3—H3	0.9500	C9—H9C	0.9800
C8—O1—C5	116.48 (11)	C5—C6—C7	119.19 (14)
C1$^{\mathrm{i}}$—C1—C2	126.23 (17)	C5—C6—H6	120.4
C1$^{\mathrm{i}}$—C1—H1	116.9	C7—C6—H6	120.4

C2—C1—H1	116.9	C6—C7—C2	121.30 (14)
C3—C2—C7	117.66 (14)	C6—C7—H7	119.3
C3—C2—C1	119.52 (13)	C2—C7—H7	119.3
C7—C2—C1	122.81 (13)	O2—C8—O1	122.30 (14)
C4—C3—C2	121.62 (14)	O2—C8—C9	126.94 (14)
C4—C3—H3	119.2	O1—C8—C9	110.76 (13)
C2—C3—H3	119.2	C8—C9—H9A	109.5
C5—C4—C3	118.61 (14)	C8—C9—H9B	109.5
C5—C4—H4	120.7	H9A—C9—H9B	109.5
C3—C4—H4	120.7	C8—C9—H9C	109.5
C4—C5—C6	121.62 (13)	H9A—C9—H9C	109.5
C4—C5—O1	119.59 (13)	H9B—C9—H9C	109.5
C6—C5—O1	118.75 (13)		
C1^{i}—C1—C2—C3	165.70 (18)	C8—O1—C5—C6	−85.05 (16)
C1^{i}—C1—C2—C7	−13.8 (3)	C4—C5—C6—C7	0.0 (2)
C7—C2—C3—C4	0.6 (2)	O1—C5—C6—C7	−177.66 (13)
C1—C2—C3—C4	−178.87 (13)	C5—C6—C7—C2	0.3 (2)
C2—C3—C4—C5	−0.4 (2)	C3—C2—C7—C6	−0.6 (2)
C3—C4—C5—C6	0.1 (2)	C1—C2—C7—C6	178.93 (13)
C3—C4—C5—O1	177.71 (12)	C5—O1—C8—O2	−5.1 (2)
C8—O1—C5—C4	97.22 (15)	C5—O1—C8—C9	175.46 (12)

Symmetry code: (i) $-x, -y+1, -z$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···*Cg*1^{ii}	0.95	2.81	3.539 (2)	135

Symmetry code: (ii) $-x, y+1/2, -z+1/2$.