Table 1. *Positional parameters and their e.s.d.'s*

	x	y	z	B_{eq}(Å^2)*
O(1)	0·5304 (4)	−0·1396 (3)	0·9213 (1)	6·89 (7)
O(2)	0·9368 (3)	0·1995 (3)	1·0045 (1)	6·11 (7)
N	0·8147 (4)	0·0757 (4)	0·9753 (1)	4·87 (7)
C(1)	0·3658 (4)	0·1088 (4)	0·8750 (1)	3·62 (7)
C(2)	0·5169 (4)	0·0113 (4)	0·9149 (1)	4·21 (8)
C(3)	0·6583 (4)	0·1335 (4)	0·9476 (1)	3·93 (7)
C(4)	0·5871 (5)	0·3073 (4)	0·9393 (1)	4·61 (8)
C(5)	0·7031 (6)	0·4269 (6)	0·9096 (3)	9·9 (1)
C(6)	0·6084 (8)	0·4498 (7)	0·8502 (2)	13·9 (1)
C(7)	0·4250 (6)	0·3528 (5)	0·8378 (2)	6·2 (1)
C(8)	0·4283 (5)	0·1694 (4)	0·8185 (1)	4·93 (9)
C(9)	0·3923 (5)	0·2956 (4)	0·8974 (1)	3·79 (7)
C(10)	0·2430 (6)	0·3813 (6)	0·9208 (2)	7·9 (1)
C(11)	0·1685 (5)	0·0375 (4)	0·8648 (1)	4·62 (8)
C(12)	0·1300 (5)	−0·1421 (4)	0·8428 (2)	5·52 (9)
C(13)	−0·0748 (7)	−0·1799 (6)	0·8336 (2)	7·9 (1)
C(14)	0·1959 (8)	−0·1802 (5)	0·7871 (2)	7·7 (1)

* B_{eq} is defined as $\frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta) \times B(1,3) + bc(\cos\alpha)B(2,3)]$.

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(1)—C(2)	1·492 (2)	C(7)—C(8)	1·533 (3)
C(2)—O(1)	1·215 (2)	C(7)—C(9)	1·531 (3)
C(2)—C(3)	1·499 (2)	C(8)—C(1)	1·567 (2)
C(3)—N	1·268 (2)	C(9)—C(1)	1·576 (2)
N—O(2)	1·401 (2)	C(9)—C(10)	1·496 (3)
C(3)—C(4)	1·479 (2)	C(1)—C(11)	1·522 (3)
C(4)—C(5)	1·543 (3)	C(11)—C(12)	1·526 (3)
C(4)—C(9)	1·538 (3)	C(12)—C(13)	1·499 (3)
C(5)—C(6)	1·412 (4)	C(12)—C(14)	1·516 (3)
C(6)—C(7)	1·522 (4)		
C(1)—C(2)—O(1)	128·42 (16)	C(4)—C(9)—C(10)	113·12 (16)
C(1)—C(2)—C(3)	107·89 (15)	C(4)—C(9)—C(1)	107·09 (14)
O(1)—C(2)—C(3)	123·70 (16)	C(8)—C(7)—C(9)	89·93 (13)
C(2)—C(3)—N	117·58 (16)	C(7)—C(8)—C(1)	91·11 (14)
C(3)—N—O(2)	113·24 (15)	C(7)—C(9)—C(1)	90·87 (12)
C(2)—C(3)—C(4)	111·12 (15)	C(1)—C(9)—C(10)	120·50 (16)
N—C(3)—C(4)	131·26 (16)	C(8)—C(1)—C(2)	111·42 (14)
C(3)—C(4)—C(5)	115·09 (18)	C(8)—C(1)—C(9)	87·05 (12)
C(3)—C(4)—C(9)	105·99 (14)	C(2)—C(1)—C(9)	105·61 (13)
C(9)—C(4)—C(5)	106·33 (16)	C(8)—C(1)—C(11)	115·71 (14)
C(4)—C(5)—C(6)	108·75 (21)	C(2)—C(1)—C(11)	116·60 (14)
C(5)—C(6)—C(7)	109·54 (22)	C(9)—C(1)—C(11)	116·49 (15)
C(6)—C(7)—C(8)	117·9 (3)	C(1)—C(11)—C(12)	120·00 (17)
C(6)—C(7)—C(9)	107·20 (18)	C(11)—C(12)—C(13)	109·86 (20)
C(7)—C(9)—C(10)	118·27 (18)	C(11)—C(12)—C(14)	113·85 (16)
C(4)—C(9)—C(7)	103·97 (16)	C(13)—C(12)—C(14)	110·00 (18)

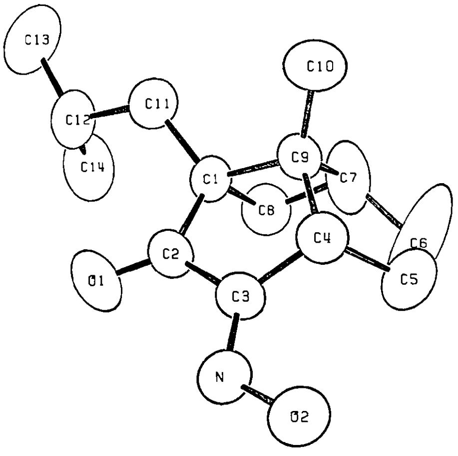

Fig. 1. View of the $C_{14}H_{21}NO_2$ molecule with the crystallographic-numbering scheme.

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(*E*)- and (*Z*)-2,3-Bis(2-methoxyphenyl)-2-butene, $C_{18}H_{20}O_2$

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Abstract. (*E*)-Isomer, $M_r = 268{\cdot}4$, monoclinic, $C2/c$, $a = 21{\cdot}096$ (5), $b = 7{\cdot}473$ (2), $c = 10{\cdot}247$ (2) Å, $\beta = 110{\cdot}41$ (2)°, $V = 1514$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}177$ (1) g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}81$ cm^{-1}, $F(000) = 576$, $T = 298$ K, final $R = 0{\cdot}042$ for 461 observations. The molecule has $\bar{1}$ symmetry, with a dihedral angle of 68·6° between the aromatic plane and the plane of the double bond. (*Z*)-Isomer, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}307$ (3), $b = 11{\cdot}053$ (3), $c = 16{\cdot}854$ (3) Å, $V = 1547$ (1) Å^3, $Z = 4$, $D_x =$

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1·152 (1) g cm^{-3}, μ(Mo $K\alpha$) = 0·80 cm^{-1}, T = 300 K, final R = 0·050 for 743 observations. The molecule has approximate symmetry 2, with the aromatic rings forming dihedral angles of 71·6 and 68·2° with the plane of the double bond.

Introduction. As part of a program to design and synthesize new molecules for the study of intramolecular reactions, considerable effort has been extended in these laboratories (Tirado-Rives, Gandour & Fronczek, 1982) in the preparation of 1,2-diphenylethene (stilbene) and 2,3-diphenyl-2-butene (dimethylstilbene) derivatives. To facilitate assignment of geometric isomers in the dimethylstilbene series, the title compounds have been synthesized and their structures have been determined. These crystal structures and others (Tirado-Rives, Oliver, Fronczek & Gandour, 1984) serve as benchmarks for NMR spectroscopic assignment of (*E*)- and (*Z*)-isomers for non-crystalline derivatives.

Experimental. Title compounds prepared by reaction of 2-methoxyacetophenone with $TiCl_4$/Zn in dioxane; this produced a mixture of the two isomers. The (*E*)-isomer spontaneously crystallized (m.p. 387–388·5 K) from hexane but the (*Z*)-isomer had to be purified by column chromatography (silica gel, hexane/CH_2Cl_2) before crystallization (m.p. 363·5–364 K) from methanol/water could be effected.

(*a*) (*E*)-Isomer: crystal size 0·22 × 0·34 × 0·52 mm. Space-group determination by systematic absences hkl with $h+k$ odd, $h0l$ with l odd, and successful refinement in centrosymmetric space group. Cell dimensions determined from setting angles of 25 reflections having $\theta > 13°$. Data collection carried out on an Enraf-Nonius CAD-4 diffractometer with graphite monochromator, by ω–2θ scans designed to yield $I \simeq 20\sigma(I)$. Scan rates varied 1·0–20·0° min^{-1}. Data having $h+k$ even, $1 < \theta < 20°$, in quadrant $h,k,\pm l$ (h 0–20, k 0–7, l –9–8) were measured, corrected for background, Lorentz, and polarization effects; equivalent data averaged ($R_{\rm int}$ = 0·020) giving 702 unique reflections. Crystal decay and absorption negligible. Three standard reflections (400, 020, 002), 1·5% intensity variation. Structure solved using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), and refined by full-matrix least squares based upon F, using 461 data for which $I > 3\sigma(I)$, and weights $w = \sigma^{-2}(F_o)$, with *Enraf–Nonius SDP* (Frenz & Okaya, 1980). Scattering factors those of Cromer & Waber (1974). Non-H atoms anisotropic; H atoms located by ΔF synthesis and included as fixed contributions with B = 5·0 Å^2. Final R = 0·042, R_w = 0·048, S = 1·315 for 91 variables. Max. shift 0·01σ in final cycle, largest residual density 0·15 e Å^{-3}.

(*b*) (*Z*)-Isomer: data collection, solution, and refinement carried out in a fashion identical to that of (*E*)-isomer. Particulars are: crystal size 0·28 × 0·36 × 0·44 mm; absences $h00$ with h odd, $0k0$ with k odd, $00l$ with l odd; unit-cell reflections $\theta > 13·5°$, one octant of data, $+h$, $+k$, $+l$ (h 0–7, k 0–10, l 0–16) with $1 < \theta < 20°$ (860 unique, 743 observed reflections), three standards (200, 020, 006), 1·0% intensity variation; B = 6·0 Å^2 for H; R = 0·050, R_w = 0·055, S = 1·203 for 181 variables, max. shift 0·035, max. density 0·22 e Å^{-3}.

Discussion. The atomic parameters for both isomers are given in Table 1.*

The (*E*)-isomer, Fig. 1, lies on a crystallographic center of symmetry, and thus has its methoxy groups in the *anti* configuration. The phenyl ring is planar, and the methoxy group lies essentially in this plane, with C(1)—C(2)—O(1)—C(9) torsion angle 180·0 (6)°. The six atoms comprising the 'plane' of the double bond C(7)=C(7′) all lie within 0·020 (4) Å of a common plane, which forms a dihedral angle of 68·6° with the aromatic ring. This dihedral angle differs markedly from the two independent values of 5·0° (Bernstein,

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters for the non-*H *atoms*

	x	y	z	$B_{\rm eq}$*(Å^2)
(*E*)-Isomer				
O(1)	0·4073 (1)	0·1978 (4)	0·5646 (3)	5·23 (8)
C(1)	0·4193 (2)	0·3762 (5)	0·3874 (4)	3·9 (1)
C(2)	0·3783 (2)	0·2625 (6)	0·4317 (4)	4·2 (1)
C(3)	0·3125 (2)	0·2229 (6)	0·3437 (5)	5·4 (1)
C(4)	0·2884 (2)	0·2940 (7)	0·2119 (5)	5·9 (1)
C(5)	0·3278 (2)	0·4037 (7)	0·1657 (5)	5·7 (1)
C(6)	0·3928 (2)	0·4430 (6)	0·2541 (5)	4·9 (1)
C(7)	0·4905 (2)	0·4182 (5)	0·4770 (4)	3·8 (1)
C(8)	0·5375 (2)	0·2617 (7)	0·4999 (5)	5·5 (1)
C(9)	0·3688 (2)	0·0816 (7)	0·6171 (5)	6·7 (1)
(*Z*)-Isomer				
O(1)	1·1428 (6)	−0·0300 (5)	0·1128 (4)	5·2 (1)
C(1)	0·8662 (9)	−0·0450 (7)	0·0874 (4)	3·4 (2)
C(2)	1·0026 (10)	0·0305 (7)	0·0960 (4)	3·9 (2)
C(3)	0·9896 (11)	0·1553 (7)	0·0887 (5)	4·4 (2)
C(4)	0·8407 (12)	0·2073 (7)	0·0731 (5)	5·1 (2)
C(5)	0·7067 (10)	0·1359 (7)	0·0651 (5)	4·5 (2)
C(6)	0·7189 (9)	0·0116 (7)	0·0731 (4)	3·5 (2)
C(7)	0·8771 (9)	−0·1797 (7)	0·0914 (4)	3·4 (2)
C(8)	0·9625 (11)	−0·2354 (8)	0·0197 (5)	5·3 (2)
C(9)	1·2862 (10)	0·0383 (9)	0·1183 (6)	6·7 (3)
O(1′)	0·4839 (6)	−0·2446 (5)	0·1855 (3)	4·6 (1)
C(1′)	0·7434 (9)	−0·1813 (7)	0·2207 (4)	3·2 (2)
C(2′)	0·5798 (9)	−0·1901 (7)	0·2404 (4)	3·5 (2)
C(3′)	0·5245 (10)	−0·1428 (7)	0·3115 (5)	4·1 (2)
C(4′)	0·6268 (11)	−0·0857 (7)	0·3643 (5)	4·7 (2)
C(5′)	0·7852 (11)	−0·0748 (8)	0·3441 (5)	5·0 (2)
C(6′)	0·8439 (10)	−0·1236 (7)	0·2720 (4)	3·9 (2)
C(7′)	0·8109 (9)	−0·2416 (7)	0·1491 (4)	3·5 (2)
C(8′)	0·8039 (11)	−0·3773 (7)	0·1490 (5)	5·6 (2)
C(9′)	0·3208 (12)	−0·2714 (9)	0·2066 (6)	6·2 (3)

E.s.d.'s in the least significant digits are shown in parentheses.

* $B_{\rm eq} = \frac{4}{3}(a^2B_{11}+b^2B_{22}+c^2B_{33}+abB_{12}\cos\gamma+acB_{13}\cos\beta+bcB_{23}\cos\alpha)$.

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* Tables of H-atom coordinates, anisotropic thermal parameters and structure-factor amplitudes for both isomers have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38995 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

1975) and 3·3° (Finder, Newton & Allinger, 1974) in (*E*)-stilbene but is close to that in (*E*)-dimethylstilbene (Valle, Busetti & Galiazzo, 1981), 74·2°.

The (*Z*)-isomer, Fig. 2, approximates symmetry 2, with the methoxy groups also in the *anti* configuration. The molecule exhibits a slight twist about the double bond [C(1)—C(7)—C(7′)—C(1′) torsion angle −9·8 (9)°]. The two phenyl rings are planar, and form dihedral angles of 71·6° (unprimed) and 68·2° (primed) with the best plane of the six atoms associated with the double bond. The methoxy groups are almost coplanar with the aromatic rings, as shown by torsion angles 176·8 (9)° C(1)—C(2)—O(1)—C(9) and 171·5 (9)° (primed).

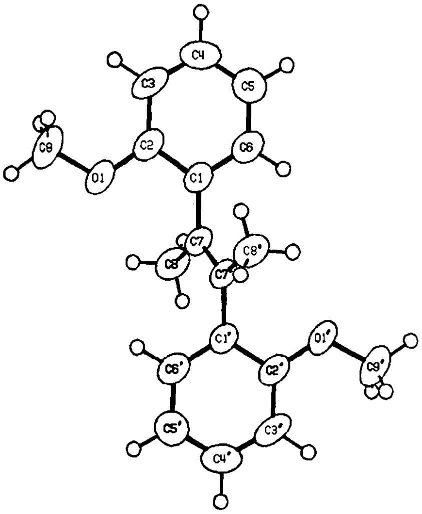

Fig. 1. *ORTEP*II (Johnson, 1976) drawing of (*E*)-2,3-bis(2-methoxyphenyl)-2-butene.

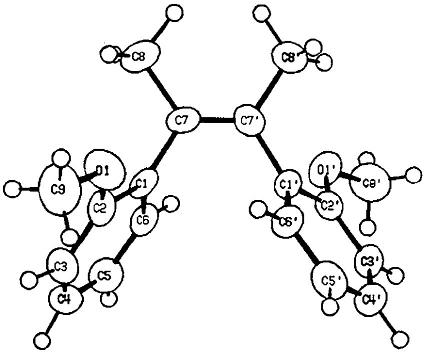

Fig. 2. *ORTEP*II drawing of (*Z*)-2,3-bis(2-methoxyphenyl)-2-butene.

Table 2. *Selected distances* (Å) *and angles* (°)

Those for the (*Z*) compound have been averaged over the two halves of the molecule.

	(*E*)-Isomer	(*Z*)-Isomer
O(1)—C(2)	1·372 (5)	1·368 (7)
O(1)—C(9)	1·417 (5)	1·423 (7)
C(1)—C(2)	1·397 (5)	1·408 (8)
C(1)—C(6)	1·377 (5)	1·378 (8)
C(1)—C(7)	1·494 (5)	1·492 (8)
C(2)—C(3)	1·398 (6)	1·387 (8)
C(3)—C(4)	1·373 (6)	1·386 (9)
C(4)—C(5)	1·365 (6)	1·368 (9)
C(5)—C(6)	1·385 (6)	1·399 (8)
C(7)—C(7′)	1·321 (6)	1·310 (10)
C(7)—C(8)	1·497 (6)	1·516 (8)
C(2)—O(1)—C(9)	118·5 (4)	118·2 (5)
C(2)—C(1)—C(6)	117·3 (4)	117·8 (5)
C(2)—C(1)—C(7)	122·1 (4)	122·0 (5)
C(6)—C(1)—C(7)	120·5 (4)	120·1 (5)
C(1)—C(2)—O(1)	115·1 (4)	115·2 (5)
C(3)—C(2)—O(1)	124·4 (4)	124·3 (6)
C(1)—C(2)—C(3)	120·5 (4)	120·5 (6)
C(1)—C(7)—C(7′)	121·9 (4)	121·8 (5)
C(1)—C(7)—C(8)	113·6 (4)	114·5 (6)
C(8)—C(7)—C(7′)	124·3 (4)	123·6 (5)

The bond distances and angles in Table 2 exhibit no significant differences between the two isomers. In particular, no detectible differences in bond angles about C(7) exist between (*Z*)- *vs* (*E*)- methyl groups. Methyl substitution at C(7) does, however, cause closure of the C(1)—C(7)—C(7′) angle, as compared to unsubstituted stilbenes. The corresponding angle in (*E*)-stilbene is 126·8 (2)° (Finder *et al.*, 1974; Bernstein, 1975) and in a macrocyclic derivative is 127·7 (4)° (Tirado-Rives *et al.*, 1982). No unusually short intermolecular contacts exist.

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