

Fig. 1. Perspective view of the ellipticine *N*-oxide molecule showing the atom labeling and the hydrogen bonding with water molecules (open bonds).

Except for a shortening of the C(15)–C(18) and C(3)–C(4) bonds as well as the C(methyl)–C(ring) bonds, the structural parameters compare well with those reported for the parent ellipticine (Courseille, Busetta & Hospital, 1974) and 5,6,11-trimethylpyrido[4,3-*b*]carbazole (Courseille, Busetta & Précigoux, 1981). The N–O bond distance and associated bond angles are comparable with those found for pyridine *N*-oxide (Desiderato & Terry, 1970). However, the N–O distance of 1.347 (4) Å is longer than those found in compounds such as 2-[(hydroxymethoxy)methyl]thio}pyridine 1-oxide [1.319 (5) Å] (Haugwitz, Toeplitz & Gougoutas, 1980), 6-chloro-3-ethoxycarbonyl-2-methylquinoxaline 1,4-dioxide [1.294 (2) and 1.308 (2) Å] (MacDonald & Arora, 1981) and others. The *N*-oxide bond length is apparently highly sensitive to its chemical environment.

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### (*E*)-1,2-Bis(2-methoxyphenyl)ethene,\* C<sub>16</sub>H<sub>16</sub>O<sub>2</sub>

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**Abstract.**  $M_r = 240.4$ , orthorhombic, *Pbca*,  $a = 13.341$  (3),  $b = 14.412$  (4),  $c = 13.753$  (4) Å,  $V = 2644$  (2) Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 1.207$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 0.73$  cm<sup>-1</sup>,  $F(000) = 1024$ ,  $T = 299$  K, final  $R = 0.044$  for 946 observations. The compound was synthesized by reductive coupling of 2-methoxybenzaldehyde. In the crystal, the molecule deviates

While the hydrogen-bond parameters\* are normal, their presence indicates that the *N*-oxide should be held strongly to DNA by hydrogen bonding. Moreover if the *N*-oxide carries such waters with it between the strands of DNA, the unwinding angle of the genetic material should be larger than that realized with ellipticine itself thus promoting higher biological activity (Sainsbury, 1979).

\* See previous footnote.

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slightly from the planar structure of *2/m* symmetry. One phenyl ring is nearly coplanar with the double bond [dihedral angle 2.7 (3)°], while the other forms a dihedral angle of 15.0 (3)° with it.

**Introduction.** In the course of our studies utilizing 1,2-bis(2-substituted-phenyl)ethenes as models for intramolecular reactions (Tirado-Rives, Gandour & Fronczek, 1982; Tirado-Rives, Oliver, Fronczek & Gandour, 1984; Fronczek, Oliver & Gandour, 1984),

\* Alternative nomenclature: (*E*)-2,2'-dimethoxystilbene.

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the title compound has been synthesized and its crystal structure has been determined. This crystal structure serves as a reference for comparison with more complex analogues.

**Experimental.** Title compound prepared by reductive coupling of 2-methoxybenzaldehyde with  $\text{TiCl}_4/\text{Zn}$  in dioxane; the reaction quantitatively afforded the desired compound. Crystals prepared from dichloromethane/petroleum ether [m.p. 411.5–412.5 K; lit. values: 413 K (Bergman & Japke, 1947), 408–409 K (Perregaard, Thomsen & Lawesson, 1975)].

Prismatic crystals, size  $0.16 \times 0.24 \times 0.64$  mm; space-group determination by systematic absences  $0kl$  with  $k$  odd,  $h0l$  with  $l$  odd, and  $hk0$  with  $h$  odd. Cell dimensions from setting angles of 25 reflections with  $13 > \theta > 11^\circ$ . Data collection on Enraf–Nonius CAD-4 diffractometer, graphite monochromator,  $\omega$ – $2\theta$  scans designed to yield  $I \approx 50\sigma(I)$ . Scan rates  $0.33$ – $4.0^\circ \text{ min}^{-1}$ . Data having  $1 < \theta < 23^\circ$  (max.  $\sin\theta/\lambda = 0.549 \text{ \AA}^{-1}$ ),  $h$  0–14,  $k$  0–15,  $l$  0–15 measured and corrected for background, Lorentz and polarization effects; 1832 unique reflections. Crystal decay and absorption negligible. Three standard reflections (400, 041, 004), 2.3% intensity variation.

Structure solved using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), refined by full-matrix least squares based upon  $F$ , using data for which  $I > 3\sigma(I)$ , weights  $w = \sigma^{-2}(F_o)$ , with *Enraf–Nonius SDP* (Frenz & Okaya, 1980), 886 unobserved data. Scattering factors those of Cromer & Waber (1974). Non-H atoms anisotropic; H atoms from  $\Delta F$  synthesis, included as fixed contributions,  $B = 5.0 \text{ \AA}^2$ . Secondary-extinction coefficient  $3.5(7) \times 10^{-7} \text{ mm}^{-1}$ . Final  $R = 0.044$ ,  $R_w = 0.055$ ,  $S = 1.525$  for 154 variables. Max. shift  $0.17\sigma$  in final cycle, max. residual density  $0.14 \text{ e \AA}^{-3}$ , min.  $0.23 \text{ e \AA}^{-3}$ .

**Discussion.** Atomic coordinates and equivalent isotropic thermal parameters for non-H atoms are given in Table 1.\*

Although the molecule (Fig. 1) exhibits only small deviations from local  $2/m$  symmetry, it is crystallographically asymmetric, unlike *(E)*-1,2-diphenylethene (Finder, Newton & Allinger, 1974) and *(E)*-1,2-bis(2-hydroxyphenyl)ethene (Tirado-Rives *et al.*, 1984), both of which exhibit  $\bar{1}$  symmetry in the crystal. Both phenyl rings are planar, as is the C=C double bond [C(1)–C(7)–C(8)–C(9) torsion angle  $-179.6(5)^\circ$ ]. The C(1) phenyl ring forms a dihedral

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

E.s.d.'s in the least-significant digits are shown in parentheses.  $B_{\text{eq}} = \frac{1}{3}(a^2B_{11} + b^2B_{22} + c^2B_{33} + abB_{12} \cos\gamma + acB_{13} \cos\beta + bcB_{23} \cos\alpha)$ .

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{eq}}(\text{\AA}^2)$
O(1)	0.5749 (2)	0.5508 (2)	0.3452 (2)	6.09 (6)
O(2)	0.6560 (2)	0.2306 (2)	–0.0007 (2)	4.92 (6)
C(1)	0.5185 (2)	0.4945 (2)	0.1957 (2)	3.54 (7)
C(2)	0.5007 (3)	0.5504 (2)	0.2767 (2)	4.35 (8)
C(3)	0.4128 (3)	0.6008 (2)	0.2859 (3)	4.97 (9)
C(4)	0.3414 (3)	0.5959 (2)	0.2139 (3)	5.20 (9)
C(5)	0.3563 (3)	0.5421 (3)	0.1328 (3)	4.90 (9)
C(6)	0.4437 (3)	0.4917 (2)	0.1248 (2)	4.21 (8)
C(7)	0.6122 (3)	0.4427 (2)	0.1876 (2)	3.86 (8)
C(8)	0.6336 (2)	0.3737 (2)	0.1279 (2)	4.05 (8)
C(9)	0.7275 (2)	0.3228 (2)	0.1217 (2)	3.47 (7)
C(10)	0.7381 (2)	0.2489 (2)	0.0570 (2)	3.77 (7)
C(11)	0.8252 (3)	0.1967 (2)	0.0528 (3)	4.55 (8)
C(12)	0.9050 (3)	0.2202 (3)	0.1123 (3)	5.28 (9)
C(13)	0.8973 (3)	0.2927 (2)	0.1761 (3)	5.04 (9)
C(14)	0.8099 (3)	0.3435 (2)	0.1801 (3)	4.59 (8)
C(15)	0.5679 (4)	0.6127 (3)	0.4233 (3)	9.3 (1)
C(16)	0.6660 (3)	0.1651 (3)	–0.0763 (3)	7.3 (1)

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

O(1)–C(2)	1.366 (4)	C(5)–C(6)	1.378 (4)
O(1)–C(15)	1.400 (4)	C(7)–C(8)	1.320 (4)
O(2)–C(10)	1.379 (3)	C(8)–C(9)	1.454 (4)
O(2)–C(16)	1.412 (4)	C(9)–C(10)	1.395 (4)
C(1)–C(2)	1.395 (4)	C(9)–C(14)	1.395 (4)
C(1)–C(6)	1.396 (4)	C(10)–C(11)	1.384 (4)
C(1)–C(7)	1.461 (4)	C(11)–C(12)	1.385 (4)
C(2)–C(3)	1.385 (4)	C(12)–C(13)	1.368 (4)
C(3)–C(4)	1.376 (5)	C(13)–C(14)	1.377 (4)
C(4)–C(5)	1.373 (4)		
C(2)–O(1)–C(15)	118.9 (3)	C(1)–C(7)–C(8)	128.1 (3)
C(10)–O(2)–C(16)	118.5 (3)	C(7)–C(8)–C(9)	127.0 (3)
C(2)–C(1)–C(6)	117.0 (3)	C(8)–C(9)–C(10)	120.7 (3)
C(2)–C(1)–C(7)	120.1 (3)	C(8)–C(9)–C(14)	122.6 (3)
C(6)–C(1)–C(7)	122.9 (3)	C(10)–C(9)–C(14)	116.7 (3)
O(1)–C(2)–C(1)	115.5 (3)	O(2)–C(10)–C(9)	115.6 (3)
O(1)–C(2)–C(3)	123.2 (3)	O(2)–C(10)–C(11)	122.6 (3)
C(1)–C(2)–C(3)	121.3 (3)	C(9)–C(10)–C(11)	121.8 (3)
C(2)–C(3)–C(4)	119.6 (3)	C(10)–C(11)–C(12)	119.1 (3)
C(3)–C(4)–C(5)	120.9 (3)	C(11)–C(12)–C(13)	120.5 (3)
C(4)–C(5)–C(6)	119.0 (3)	C(12)–C(13)–C(14)	119.7 (3)
C(1)–C(6)–C(5)	122.3 (3)	C(9)–C(14)–C(13)	122.1 (3)

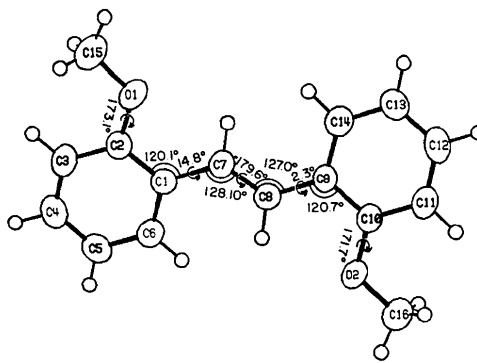


Fig. 1. *(E)*-1,2-Bis(2-methoxyphenyl)ethene with selected torsion and bond angles. E.s.d.'s for torsion angles are  $0.5^\circ$ .

\* Lists of H-atom coordinates, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39585 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

angle of  $15.0(3)^\circ$  with the double bond, quite similar to the value of  $17.6^\circ$  in (*E*)-1,2-bis(2-hydroxyphenyl)ethene. The C(9) phenyl ring forms a dihedral angle of  $2.7(3)^\circ$  with the double bond, a value much closer to that of (*E*)-1,2-diphenylethene ( $3.3^\circ$ ) than to those of the 2-substituted phenyl derivatives.

Bond distances and angles are given in Table 2. Of note are the C(7)–C(8) double-bond distance of  $1.320(4) \text{ \AA}$ , and the angles at the two olefinic atoms. The chemically equivalent angles C(1)–C(7)–C(8),  $128.1(3)^\circ$ , and C(7)–C(8)–C(9),  $127.0(3)^\circ$ , are slightly larger than that found in (*E*)-1,2-bis(2-hydroxyphenyl)ethene,  $125.2(3)^\circ$ , but not significantly different from that found in (*E*)-1,2-diphenylethene,  $126.7(2)^\circ$ . The methoxy substituents are slightly non-coplanar with the phenyl rings, described by torsion angles C(1)–C(2)–O(1)–C(15),  $173.1(5)^\circ$ , and C(9)–C(10)–O(2)–C(16),  $171.7(5)^\circ$ . This contrasts with (*E*)-2,3-bis(2-methoxyphenyl)-2-butene, in which the corresponding torsion angle is  $180.0(6)^\circ$ , but compares well with (*Z*)-2,3-bis(2-methoxyphenyl)-2-butene, in which the two independent torsion angles are  $176.8(9)$  and  $171.5(9)^\circ$  (Fronczek *et al.*, 1984).

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### (*Z*)-2,3-Diphenyl-2-butene,\* $C_{16}H_{16}$

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**Abstract.**  $M_r = 208.3$ , monoclinic,  $P2_1/n$ ,  $a = 10.217(3)$ ,  $b = 8.703(1)$ ,  $c = 14.342(4) \text{ \AA}$ ,  $\beta = 95.67(2)^\circ$ ,  $V = 1269.0(9) \text{ \AA}^3$ ,  $Z = 4$ ,  $D_x = 1.090 \text{ g cm}^{-3}$ ,  $\lambda = 0.71073 \text{ \AA}$ ,  $\mu(\text{Mo } K\alpha) = 0.57 \text{ cm}^{-1}$ ,  $F(000) = 448$ ,  $T = 296 \text{ K}$ ,  $R = 0.050$  for 998 observations (of 1981 unique data). The molecule approximates symmetry 2, with the two phenyl rings forming dihedral angles of  $59.1$  and  $49.7^\circ$  with the best plane of the double bond. The double bond is twisted slightly away from planarity.

**Introduction.** In the course of our studies utilizing 2,3-bis(2-substituted-phenyl)-2-butenes as models for intramolecular reactions (Tirado-Rives, Oliver, Fronczek & Gandour, 1984; Fronczek, Oliver & Gandour,

1984), the crystal structure of the title compound has been determined. This crystal structure serves as a reference for comparison with more complex analogues and complements the recent report (Valle, Busetti & Galiazzo, 1981) of the crystal structure of the (*E*) isomer.

**Experimental.** Title compound prepared by a previously published procedure (Mukaiyama, Sato & Hanna, 1973), which produced a mixture of isomers. During removal under vacuum of traces of solvent from the mixture, the (*Z*) isomer sublimed. A suitable crystal was obtained from the sublimed material, m.p.  $338 \text{ K}$  [lit. m.p.  $340 \text{ K}$  (von Wessely & Welleba, 1941)]. Crystal  $0.32 \times 0.52 \times 0.60 \text{ mm}$  mounted in a thin-walled glass capillary to prevent sublimation. Space group from systematic absences  $0k0$  with  $k$  odd,  $h0l$  with  $h + l$  odd. Cell dimensions determined by least

\* Alternative nomenclature: (*Z*)- $\alpha,\beta$ -dimethylstilbene.

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