

molecule is bonded to two vicinal ones by eight hydrogen bonds in compound (1) and to four neighbors by ten hydrogen bonds in compound (2) (distances in Table 3). This is a general feature of these compounds (Satzke & Mackay, 1975; Sindt & Mackay, 1977; Darbon *et al.*, 1984) as well as of carbohydrates (Gatehouse & Poppleton, 1971). Preliminary qualitative tests showed compounds (1) and (2) to be respectively neutral (with a slightly sweet taste) and bitter.

We thank Mr S. Lecocq, University of Lyon I (France), for technical assistance in the data collection.

References

- BHATTACHARJEE, S. & JEFFREY, G. A. (1983). *Mol. Cryst. Liq. Cryst.* **101**, 247–260.
- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
- DARBON, N., ODDON, Y., LACOMBE, J. M., DECOSTER, E., PAVIA, A. A. & REBOUL, J. P. (1984). *Acta Cryst.* **C40**, 1105–1107.
- GATEHOUSE, B. M. & POPPLETON, B. J. (1971). *Acta Cryst.* **B27**, 10–20.
- ISHIKAWA, T. (1964). *Nippon Kagaku Zasshi*, **85**, 897–900.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univ. of York, England, and Louvain, Belgium.
- SATZKE, L. O. G. & MACKAY, M. F. (1975). *Acta Cryst.* **B31**, 1128–1132.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- SINDT, A. C. & MACKAY, M. F. (1977). *Acta Cryst.* **B33**, 2659–2662.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1985). **C41**, 1327–1329

(*E*)-1,2-Bis(2-methylphenyl)ethene,* C₁₆H₁₆

BY JULIAN TIRADO-RIVES, FRANK R. FRONCZEK AND RICHARD D. GANDOUR†

Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803-1804, USA

(Received 14 February 1985; accepted 2 May 1985)

Abstract. $M_r = 208.3$, monoclinic, $P2_1/n$, $a = 8.680$ (2), $b = 6.687$ (2), $c = 10.886$ (2) Å, $\beta = 98.55$ (2)°, $V = 624.8$ (5) Å³, $Z = 2$, $D_x = 1.107$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.58$ cm⁻¹, $F(000) = 224$, $T = 299$ K, $R = 0.044$ for 658 observations. The molecule has $\bar{1}$ symmetry in the crystal. The phenyl ring is twisted by 10.7 (3)° out of the plane of the double bond. The olefinic C=C distance is unusually short, 1.284 (4) Å.

Introduction. As part of our continuing studies on the structures of 1,2-bis(2-substituted-phenyl)ethenes (Tirado-Rives, Oliver, Fronczek & Gandour, 1984; Fronczek, Oliver & Gandour, 1984; Jungk, Fronczek & Gandour, 1984) as possible frameworks for models of intramolecular reactions, the title compound's crystal structure has been determined. This crystal structure serves as a reference for comparison with other members of the series as well as with recently reported data (Kobayashi, Suzuki & Ogawa, 1982) on its conformation in the gas phase and in solution.

Experimental. Title compound isolated chromatographically from a mixture produced in the reaction of 2-hydroxybenzaldehyde, 2-methylbenzaldehyde and TiCl₄/Zn, recrystallized from hexane, m.p. 356 K, crystal size 0.16 × 0.28 × 0.32 mm. Space group from systematic absences $h0l$ with $h+l$ odd and $0k0$ with k odd; cell dimensions from setting angles of 25 reflections having $13 > \theta > 11^\circ$. Data collected on Enraf-Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation, graphite monochromator, $\omega-2\theta$ scans designed for $I = 100\sigma(I)$. Scan rates 0.19–4.0° min⁻¹. Data having $1 \leq \theta \leq 25^\circ$, $0 \leq h \leq 10$, $0 \leq k \leq 7$, $-12 \leq l \leq 12$ measured and corrected for background, decay, Lorentz and polarization effects. Absorption negligible. Three standard reflections (200, 020, 002), 8% intensity decay. 1168 data measured, 1090 unique, $R_{\text{int}} = 0.019$. Structure solved using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), refined by full-matrix least squares based upon F , using data for which $I > 1\sigma(I)$, weights $w = \sigma^{-2}(F_o)$, $\sigma(F_o^2) = [\sigma^2(I_{\text{CS}}) + (0.02I)^2]^{0.5}/Lp$, with *Enraf-Nonius SDP* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), 432 unobserved data. Non-H atoms refined anisotropically; H atoms

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

† To whom correspondence should be addressed.

located by ΔF map and refined isotropically. Final $R = 0.044$ (0.123 for all data), $wR = 0.047$, $S = 1.546$ for 106 variables. Max. shift 0.31σ in final cycle, max. residual density $0.12 \text{ e } \text{\AA}^{-3}$, min. $-0.17 \text{ e } \text{\AA}^{-3}$, extinction coefficient, $g = 3.1(6) \times 10^{-6} [|F_c| = |F_o| \times (1 + gI)]$.

Discussion. The atomic parameters are given in Table 1.* A summary of distances and angles is presented in Table 2. A view of the molecule is shown in Fig. 1.

A comparison of structural parameters for 1,2-bis(2-substituted-phenyl)ethenes is made in Table 3. Steric effects can account for the general trend of increases in φ 's and θ 's when an *ortho* H is replaced by some group X . The surprising result is the short value for d_1 in the title compound. A similarly short d_1 was observed in one of the two independent molecules of (*E*)-1,2-diphenylethene, $1.295(6) \text{ \AA}$ (Finder, Newton & Allinger, 1974), which was attributed to disorder. Residual difference-map peaks in that determination support the disorder hypothesis, but no such residual density exists in the present structure. In an independent determination of (*E*)-1,2-diphenylethene (Bernstein, 1975), the short C=C bond distance was 1.288 \AA , but no mention was made of residual density.

Comparing the title compound with an isomer (*E*)-2,3-diphenyl-2-butene (Valle, Busetti & Galiazzo, 1981) reveals considerable difference in the orientation of the aromatic rings with respect to the double bond. In the latter case, the aromatic rings are approximately orthogonal to the plane of the double bond. Methyl groups can interact with two *ortho* H's, one on each ring. The more crowded contact would be the 1,7 interaction of methyl H's with the *ortho* H in the distal aromatic ring rather than the 1,6 interaction of methyl H's with the *ortho* H on the proximal ring. The title compound contains the reverse 1,6 interaction, methyl groups *ortho* and vinyl H's. Since the aromatic rings in the title compound are just slightly twisted out of the plane of the double bond, the reason for the orthogonal conformation in (*E*)-2,3-diphenyl-2-butene is the unfavorable 1,7 interaction.

The non-planarity of the aromatic rings with the double bond observed in the crystal is also found in solution and in the gas phase. Conformational analysis studies (Kobayashi *et al.*, 1982) of stilbenes in the gas phase by photoelectron spectroscopy and in solution by electronic absorption spectroscopy give values for $\varphi_1(\varphi_3)$ of $40(5)^\circ$, and 20.2 or 37.3° , respectively. The $\varphi_1(\varphi_3)$ value of $10.7(3)^\circ$ observed in the crystal suggests that crystallization 'flattens' the molecule.

* Tables 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 42227 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Crystallographic coordinates and equivalent isotropic thermal parameters

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

	x	y	z	$B_{eq}(\text{\AA}^2)$
C(1)	0.4928 (2)	0.1356 (3)	0.1561 (2)	4.36 (4)
C(2)	0.5496 (2)	0.0957 (3)	0.2815 (2)	4.32 (4)
C(3)	0.5109 (2)	0.2264 (3)	0.3707 (2)	5.11 (5)
C(4)	0.4195 (2)	0.3908 (4)	0.3405 (2)	5.98 (5)
C(5)	0.3651 (2)	0.4326 (4)	0.2182 (2)	6.09 (6)
C(6)	0.4025 (2)	0.3063 (3)	0.1274 (2)	5.36 (5)
C(7)	0.5292 (2)	0.0011 (3)	0.0578 (2)	5.36 (5)
C(8)	0.6502 (3)	-0.0820 (4)	0.3190 (2)	6.00 (5)

Table 2. Distances (\AA) and angles ($^\circ$)

C(1)-C(2)	1.406 (2)	C(5)-C(6)	1.375 (3)
C(1)-C(6)	1.393 (2)	C(5)-H(5)	0.96 (2)
C(1)-C(7)	1.469 (2)	C(6)-H(6)	0.97 (2)
C(2)-C(3)	1.384 (2)	C(7)-C(7')	1.284 (4)
C(2)-C(8)	1.496 (3)	C(7)-H(7)	0.94 (3)
C(3)-C(4)	1.367 (3)	C(8)-H(81)	1.02 (2)
C(3)-H(3)	1.01 (2)	C(8)-H(82)	0.98 (2)
C(4)-C(5)	1.374 (3)	C(8)-H(83)	0.96 (2)
C(4)-H(4)	0.94 (2)		
C(2)-C(1)-C(6)	118.6 (2)	C(6)-C(5)-H(5)	119 (1)
C(2)-C(1)-C(7)	120.6 (2)	C(1)-C(6)-C(5)	121.8 (2)
C(6)-C(1)-C(7)	120.8 (2)	C(1)-C(6)-H(6)	119 (1)
C(1)-C(2)-C(3)	118.3 (2)	C(5)-C(6)-H(6)	120 (1)
C(1)-C(2)-C(8)	121.5 (2)	C(1)-C(7)-C(7')	128.7 (3)
C(3)-C(2)-C(8)	120.2 (2)	C(1)-C(7)-H(7)	114 (1)
C(2)-C(3)-C(4)	122.1 (2)	C(7')-C(7)-H(7)	117 (1)
C(2)-C(3)-H(3)	119 (1)	C(2)-C(8)-H(81)	112 (1)
C(4)-C(3)-H(3)	119 (1)	C(2)-C(8)-H(82)	112 (1)
C(3)-C(4)-C(5)	120.0 (2)	C(2)-C(8)-H(83)	111 (1)
C(3)-C(4)-H(4)	121 (1)	H(81)-C(8)-H(82)	103 (2)
C(5)-C(4)-H(4)	120 (1)	H(81)-C(8)-H(83)	109 (2)
C(4)-C(5)-C(6)	119.2 (2)	H(82)-C(8)-H(83)	108 (2)
C(4)-C(5)-H(5)	122 (1)		

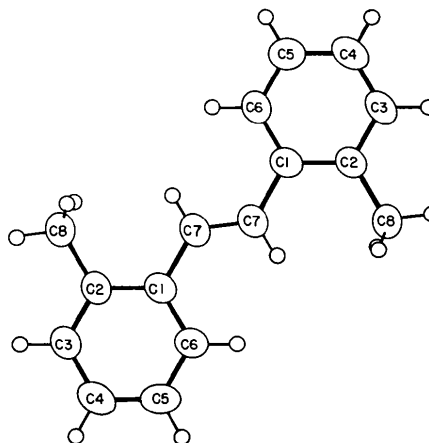
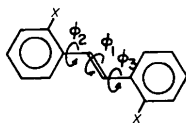
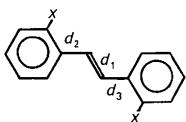


Fig. 1. Perspective drawing of (*E*)-1,2-bis(2-methylphenyl)ethene showing atom labeling.

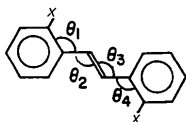
Table 3. Comparison of structural parameters for 1,2-bis(2-substituted-phenyl)ethenes



X	$\phi_1(^{\circ})$	$\phi_2(^{\circ})$	$\phi_3(^{\circ})$	Reference
Me	180	10.7 (3)	-10.7 (3)	This work
OMe	179.6 (5)	14.8 (5)	-2.3 (5)	Jungk <i>et al.</i> (1984)
OH	180	17.6 (4)	-17.6 (4)	Tirado-Rives <i>et al.</i> (1984)
H	180	5.0 (4)	-5.0 (4)	Bernstein (1975)



X	$d_1(\text{\AA})$	$d_2(\text{\AA})$	$d_3(\text{\AA})$
Me	1.284 (4)	1.469 (2)	1.469 (2)
OMe	1.320 (4)	1.461 (4)	1.454 (4)
OH	1.328 (4)	1.476 (4)	1.476 (4)
H	1.318 (3)	1.469 (4)	1.469 (4)



X	$\theta_1(^{\circ})$	$\theta_2(^{\circ})$	$\theta_3(^{\circ})$	$\theta_4(^{\circ})$
Me	120.6 (2)	128.7 (3)	128.7 (3)	120.6 (2)
OMe	120.1 (3)	128.1 (3)	127.0 (3)	120.7 (3)
OH	120.2 (3)	125.2 (3)	125.2 (3)	120.2 (3)
H	118.9 (2)	126.7 (2)	126.7 (2)	118.9 (2)

Bond distances within the aromatic ring exhibit a systematic shortening of bonds involving C(4) and C(5), which have the largest thermal parameters. This apparent shortening is probably due to librational motion, for which corrections have not been made.

Support for this work was provided by grant GM29128 from the National Institutes of Health.

References

- BERNSTEIN, J. (1975). *Acta Cryst.* **B31**, 1268–1271.
- CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- FINDER, C. J., NEWTON, M. G. & ALLINGER, N. L. (1974). *Acta Cryst.* **B30**, 411–415.
- FRENZ, B. A. & OKAYA, Y. (1980). *Enraf-Nonius Structure Determination Package*. Delft, Holland.
- FRONCZEK, F. R., OLIVER, M. A. & GANDOUR, R. D. (1984). *Acta Cryst.* **C40**, 504–506.
- JUNGK, S. J., FRONCZEK, F. R. & GANDOUR, R. D. (1984). *Acta Cryst.* **C40**, 1873–1875.
- KOBAYASHI, T., SUZUKI, H. & OGAWA, K. (1982). *Bull. Chem. Soc. Jpn.* **55**, 1734–1738.
- MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCO, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- TIRADO-RIVES, J., OLIVER, M. A., FRONCZEK, F. R. & GANDOUR, R. D. (1984). *J. Org. Chem.* **49**, 1627–1634.
- VALLE, G., Busetti, V. & GALIAZZO, G. (1981). *Cryst. Struct. Commun.* **10**, 867–870.

Acta Cryst. (1985). **C41**, 1329–1331

Structure of 3,5-Bis(diphenylamino)-1,2,4-thiadiazole, $C_{26}H_{20}N_4S$

BY HITOSHI SENDA AND JURO MARUHA

The College of Liberal Arts, Kanazawa University, Kanazawa 920, Japan

(Received 14 March 1985; accepted 15 May 1985)

Abstract. $M_r = 420.5$, triclinic, $P\bar{1}$, $a = 9.741$ (3), $b = 13.232$ (3), $c = 9.631$ (2) Å, $\alpha = 110.27$ (1), $\beta = 109.36$ (2), $\gamma = 71.96$ (2)°, $V = 1071.7$ Å³, $Z = 2$, $D_m = 1.30$, $D_x = 1.300$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.167$ mm⁻¹, $F(000) = 440$, $T = 298$ K, final $R = 0.075$ for 4043 observed unique reflections. One of the four phenyl groups is weakly conjugated to the central heterocyclic ring through the exocyclic N atom. The heterocyclic ring is planar with the exocyclic N atoms deviating from this plane by -0.030 and 0.033 Å. The molecules are held together mainly by van der Waals interactions.

Introduction. During the systematic studies of oxidation of substituted thioureas by the iron(III) ion, we found that 1,1-diphenylthiourea changes to a 1,2,4-thiadiazole compound. The structure of the compound has been determined to reveal the reaction mechanism. The oxidation of 1-phenylthiourea yields a heterocyclic base (Hector, 1889) and the structure has been determined from X-ray diffraction study to be a 1,2,4-thiadiazoline derivative (Butler, Glidewell & Liles, 1978). On the other hand, oxidation of 1-alkyl-3-arylthiourea gives rise to 1,2,4-thiadiazolidine compounds, and several crystallographic investigations have been published