

Berkeley. We wish to thank Dr F. J. Hollander for his assistance and the instructors of the course, Professor K. N. Raymond, Professor D. H. Templeton and Diane Fisher. The equipment used was funded in part by the National Science Foundation, grant CHE79-007027.

References

DARWIN, C. G. (1922). *Philos. Mag.* **43**, 800–880.

FRENZ, B. A. (1982). *Enraf-Nonius Structure Determination Package*. College Station, Texas 77840, USA, and Enraf-Nonius, Delft, Holland; as revised locally by F. HOLLANDER.
International Tables for X-ray Crystallography (1974). Vol. IV, Tables 2.2B and 2.3.1. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
 MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Acta Cryst. (1985). **C41**, 1324–1327

N-Isopropyl-D-gluconamide (1), C₉H₁₉NO₆, and *N,N*-Diethyl-D-gluconamide (2), C₁₀H₂₁NO₆

BY N. DARBON-MEYSSONNIER, Y. ODDON, E. DECOSTER AND A. A. PAVIA

Laboratoire de Chimie Bioorganique, UER des Sciences, Université d'Avignon, 33 rue Louis Pasteur, 84000 Avignon, France

G. PÈPE

Centre de Recherche sur les Mécanismes de la Croissance Cristalline, Université d'Aix-Marseille III, Campus Luminy, Case 913, 13288 Marseille CEDEX 2, France

AND J. P. REBOUL

Laboratoire de Chimie Thérapeutique, Faculté de Pharmacie, Université d'Aix-Marseille II, 27 boulevard Jean Moulin, 13385 Marseille CEDEX 5, France

(Received 8 January 1985; accepted 1 May 1985)

Abstract. (1): $M_r = 237.2$, orthorhombic, $P2_12_12_1$, $a = 25.153$ (4), $b = 8.615$ (1), $c = 5.350$ (1) Å, $V = 1159.4$ (6) Å³, $Z = 4$, $D_m = 1.37$ (1), $D_x = 1.359$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 8.73$ cm⁻¹, $F(000) = 512$, $T = 293$ K, $R = 0.049$ for 585 independent observed reflections. (2): $M_r = 251.3$, orthorhombic, $P2_12_12_1$, $a = 6.870$ (1), $b = 7.655$ (1), $c = 24.029$ (4) Å, $V = 1263.7$ (6) Å³, $Z = 4$, $D_m = 1.33$ (1), $D_x = 1.321$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 8.26$ cm⁻¹, $F(000) = 544$, $T = 293$ K, $R = 0.059$ for 982 independent observed reflections. In both crystals, molecules are associated by hydrogen bonds. In the case of *N,N*-diethyl-D-gluconamide, one of the ethyl groups is found to be disordered. The analysis of this disorder gives two positions for this group, with occupancies 0.25 and 0.75 respectively. The carbonyl bond introduces dissymmetry in the bond lengths around the N atoms.

Introduction. The structural analyses of *N*-isopropyl-D-gluconamide and *N,N*-diethyl-D-gluconamide have been integrated into a general program of synthesis of

such compounds, to find the relation between conformational properties and the sweetening or souring power of these molecules.

Experimental. Compounds studied herein synthesized following the Ishikawa (1964) procedure. Single crystals obtained at room temperature, by evaporation of a methanol solution for compound (1) and an ethanol-ethoxide solution for compound (2), D_m by flotation in hexane/CCl₄. Colorless, transparent crystals [truncated sticks for (1), parallelograms for (2)]. For data collection the crystals were cut to a cubic shape [respectively 0.18 and 0.15 mm side for (1) and (2)]. Cell parameters refined using 25 reflections. Nonius CAD-4 diffractometer, graphite monochromator, θ - 2θ scan mode, aperture $(1.6 + 0.5 \text{tg}\theta)^\circ$, max. scan time 60 s, $(\sin\theta)_{\text{max}}/\lambda = 0.620$ Å⁻¹. Three standard reflections monitored every 75 reflections, 3% variation. Range of hkl : h 0→30, k 0→10, l 0→6 for (1) and h 0→8, k 0→9, l 0→29 for (2), 1328 and 1502 symmetry-independent reflections collected respectively for compound (1) and compound (2), 585 reflections in

the case of (1) and 982 reflections for (2) kept to solve and refine the structure [$I > 2.5\sigma(I)$]. Intensities corrected for Lorentz and polarization factors, no correction for absorption. Structures solved by direct methods using *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), with $E \geq 1.12$. In both cases all non-H atoms were present on the Fourier synthesis with the best figure of merit. Structures refined by full-matrix least squares using *SHELX76* (Sheldrick, 1976). Scattering factors for C and N atoms given by Cromer & Mann (1968), for H defined by Stewart, Davidson & Simpson (1965). All H atoms appear on difference Fourier maps. Anisotropic thermal refinement for non-H atoms and isotropic for H. However, the thermal parameters of one of the ethyl groups of the second compound are very high ($B_{eq} > 15 \text{ \AA}^2$). This observation indicates disorder at this position, which was analyzed as follows: the occupancy degree of the dominant position was refined by fixing the isotropic thermal parameters of the atoms C(9) and C(10) at 3 \AA^2 . Subsequent difference maps gave another position for the ethyl group. The occupancy of that position was refined in the same way but without the ethyl group previously determined; this technique revealed the occupancy degrees for this position, respectively 0.75 and 0.25. (It was not possible to locate the H atoms on the second position.) The analysis of the crystal packing indicates that this disorder may be related to the small number of contacts of this group. Fig. 1(b) and (c) shows the molecule of (2) with the ethyl group [C(9)–C(10)] in the two positions. Final R values 0.049 and 0.059 for compounds (1) and (2). $\sum w||F_o| - |F_c||^2$ minimized, $w = 1$. Max. and min. height in final $\Delta\rho$ map 0.14 and -0.09 e \AA^{-3} , $(\Delta/\sigma)_{\max} = 0.15$ for (1) and $\Delta\rho_{\max} = 0.13$, $\Delta\rho_{\min} = -0.12 \text{ e \AA}^{-3}$, $(\Delta/\sigma)_{\max} = 0.12$ for (2).

Discussion. Atomic parameters are given in Table 1,* bond distances and angles in Table 2.

The disorder of the ethyl group C(9)–C(10) complicated the analysis of its position. This feature was also observed by Bhattacharjee & Jeffrey (1983) in octyl 1-thio- β -D-xylopyranoside, in which the disorder is in the carbohydrate moiety. In the latter, two orientations of nearly equal occupancy are observed. The linearity of the gluconamide group can be observed in the figures. An intramolecular hydrogen bond $\text{N}-\text{H}\cdots\text{O}(2)$ is present in compound (1); this is also a characteristic of *N*-cyclohexyl-D-gluconamide (Darbon, Oddon, Lacombe, Decoster, Pavia & Reboul, 1984)

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and a comparison of bond lengths around the N atom in compounds (1), (2), *A* and *B* have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42230 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(compound *A*) and *N*-(2-chloroethyl)-D-gluconamide (Satzke & Mackay, 1975; Sindt & Mackay, 1977) (compound *B*). The atoms C(2), C(3), C(4), C(5), C(6) and C(7), N, C(1), C(2), O(1) of both compounds are distributed in two planes with a maximum distance to these planes of 0.04 \AA . The angle between the planes is respectively 62.7° for compound (1) and 76.3° for compound (2). These values are very close to the values observed in the above-mentioned compounds, respectively 61.7 and 69.8° . The carbonyl C(1)–O(1) introduces dissymmetry in the bond lengths around the N atoms.

Molecular packing and hydrogen bonding

The analysis of the crystal packing of these compounds shows in both cases a strong network of hydrogen bonds. Nevertheless, in *N,N*-diethyl-D-gluconamide one of the ethyl groups is found to be

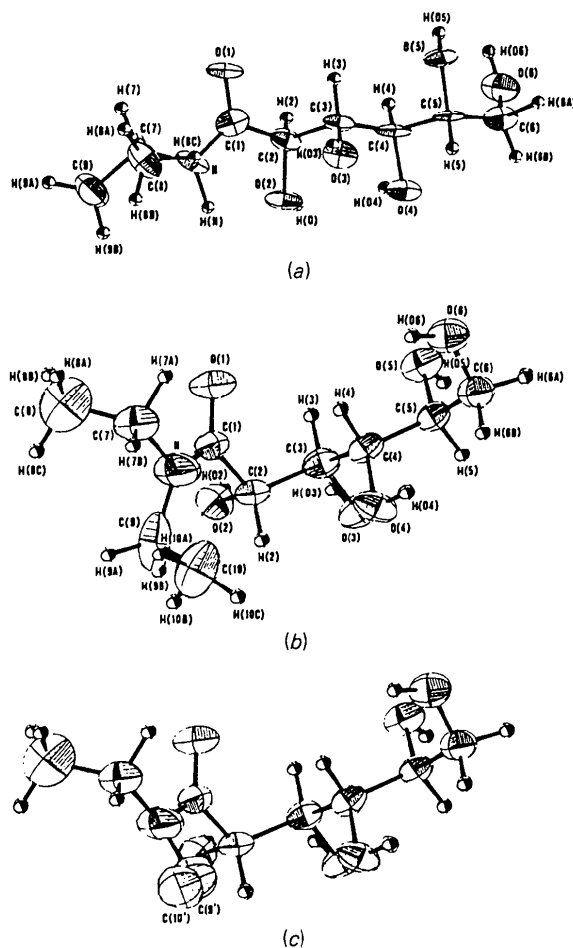


Fig. 1. ORTEP (Johnson, 1965) drawing of (a) compound (1) and (b,c) compound (2) showing atom numbering. The non-H atoms are represented by 50% probability ellipsoids while the H atoms are drawn as spheres of arbitrary size.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors for the non-H atoms for compounds (1) and (2)

$$B_{eq} = \frac{1}{3} \pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$B_{eq}/B(\text{\AA}^2)$
Compound (1)				
C(1)	6428 (5)	7252 (12)	7041 (16)	3.2 (4)
C(2)	6981 (5)	6735 (11)	6327 (15)	2.9 (4)
C(3)	7397 (5)	7778 (10)	7474 (15)	2.5 (4)
C(4)	7947 (6)	7486 (10)	6621 (18)	2.9 (5)
C(5)	8384 (5)	8479 (10)	7834 (17)	2.9 (4)
C(6)	8910 (6)	8286 (12)	6749 (19)	3.5 (4)
C(7)	5550 (5)	6558 (11)	8704 (17)	3.9 (5)
C(8)	5526 (6)	7056 (12)	11409 (19)	6.5 (5)
C(9)	5213 (9)	5047 (18)	8267 (22)	5.7 (5)
N	6096 (4)	6222 (9)	8069 (14)	3.9 (4)
O(1)	6283 (3)	8595 (8)	6469 (12)	3.7 (4)
O(2)	7053 (3)	5146 (9)	7079 (13)	3.9 (4)
O(3)	7369 (3)	7605 (8)	10155 (11)	3.8 (4)
O(4)	8136 (4)	5910 (8)	7171 (13)	3.4 (4)
O(5)	8210 (3)	10076 (7)	7523 (11)	3.2 (4)
O(6)	8952 (3)	8449 (8)	4107 (14)	5.3 (4)
Compound (2)				
C(1)	1883 (12)	3628 (10)	8768 (3)	3.6 (4)
C(2)	2291 (11)	1759 (9)	8590 (3)	3.1 (4)
C(3)	1903 (11)	1495 (9)	7978 (3)	3.4 (4)
C(4)	3053 (10)	2670 (10)	7584 (3)	3.1 (4)
C(5)	2514 (10)	2322 (9)	6976 (3)	3.0 (4)
C(6)	3382 (13)	3567 (10)	6574 (3)	3.9 (4)
C(7)	2644 (14)	6266 (9)	9261 (4)	4.8 (5)
C(8)	1286 (19)	6519 (16)	9759 (5)	8.0 (5)
C(9)	4380 (16)	3489 (10)	9471 (5)	6.6 (5)
C(10)	6395 (20)	3951 (18)	9219 (5)	7.3 (5)
C(9')	5180 (15)	4057 (10)	9008 (4)	11.1 (11)
C(10')	6663 (18)	5483 (16)	9128 (5)	10.6 (11)
N	3093 (10)	4475 (10)	9096 (3)	6.4 (4)
O(1)	424 (9)	4339 (8)	8580 (3)	4.8 (4)
O(2)	1073 (9)	661 (8)	8920 (2)	4.2 (4)
O(3)	2327 (8)	-310 (7)	7838 (2)	3.8 (4)
O(4)	5078 (8)	2452 (8)	7681 (2)	4.2 (4)
O(5)	474 (8)	2297 (8)	6920 (3)	4.2 (4)
O(6)	2667 (9)	5273 (7)	6627 (2)	4.6 (4)

disordered. We have analyzed this disorder. Projections of both crystals along the [001] axis for (1) (Fig. 2a) and along the [010] axis for (2) (Fig. 2b) show an analogous packing; molecules are shown to be parallel to the longest axis. The stability of the crystals is achieved by a network of hydrogen bonds; each

Table 2. Bond lengths (\AA) and angles ($^\circ$) for compounds (1) and (2)

	Compound (1)	Compound (2)	
C(1)–C(2)	1.510 (17)	C(1)–C(2)	1.519 (9)
C(2)–C(3)	1.510 (15)	C(2)–C(3)	1.508 (9)
C(3)–C(4)	1.478 (18)	C(3)–C(4)	1.527 (10)
C(4)–C(5)	1.537 (16)	C(4)–C(5)	1.529 (9)
C(5)–C(6)	1.454 (18)	C(5)–C(6)	1.483 (10)
C(7)–C(8)	1.511 (13)	C(7)–C(8)	1.531 (8)
C(7)–C(9)	1.571 (20)	C(9)–C(10)	1.552 (17)
C(1)–N	1.337 (14)	C(9')–C(10')	1.521 (15)
C(7)–N	1.444 (15)	C(1)–N	1.316 (10)
C(1)–O(1)	1.251 (12)	C(7)–N	1.460 (7)
C(2)–O(2)	1.438 (12)	C(9)–N	1.471 (6)
C(3)–O(3)	1.444 (9)	C(9')–N	1.484 (6)
C(4)–O(4)	1.468 (12)	C(1)–O(1)	1.227 (9)
C(5)–O(5)	1.453 (10)	C(2)–O(2)	1.428 (9)
C(6)–O(6)	1.424 (12)	C(3)–O(3)	1.451 (8)
		C(4)–O(4)	1.421 (8)
		C(5)–O(5)	1.408 (8)
		C(6)–O(6)	1.402 (8)
C(1)–C(2)–C(3)	111.1 (6)	C(1)–C(2)–C(3)	111.7 (6)
C(2)–C(3)–C(4)	114.9 (6)	C(2)–C(3)–C(4)	115.7 (6)
C(3)–C(4)–C(5)	116.4 (6)	C(3)–C(4)–C(5)	111.4 (6)
C(4)–C(5)–C(6)	114.8 (6)	C(4)–C(5)–C(6)	114.4 (6)
C(1)–N–C(7)	123.9 (6)	C(1)–N–C(7)	119.5 (6)
N–C(7)–C(8)	108.7 (6)	N–C(7)–C(8)	117.4 (8)
N–C(7)–C(9)	108.2 (7)	N–C(7)–C(9)	100.4 (3)
N–C(1)–C(2)	119.0 (6)	N–C(9')–C(10')	117.7 (3)
C(1)–C(1)–N	122.1 (6)	C(1)–N–C(9)	119.6 (6)
O(1)–C(1)–C(2)	118.7 (6)	C(1)–N–C(9')	114.8 (5)
O(2)–C(2)–C(1)	109.0 (5)	C(7)–N–C(9)	116.2 (6)
O(2)–C(2)–C(3)	111.4 (5)	C(7)–N–C(9')	116.4 (7)
O(3)–C(3)–C(2)	108.0 (5)	N–C(1)–C(2)	121.1 (7)
O(3)–C(3)–C(4)	109.5 (5)	O(1)–C(1)–N	121.2 (7)
O(4)–C(4)–C(3)	113.5 (5)	O(1)–C(1)–C(2)	117.7 (7)
O(4)–C(4)–C(5)	101.4 (5)	O(2)–C(2)–C(1)	106.8 (6)
O(5)–C(5)–C(4)	105.3 (5)	O(2)–C(2)–C(3)	111.1 (6)
O(5)–C(5)–C(6)	109.7 (6)	O(3)–C(3)–C(2)	108.5 (6)
O(6)–C(6)–C(5)	116.9 (5)	O(3)–C(3)–C(4)	108.3 (6)
C(8)–C(7)–C(9)	110.9 (8)	O(4)–C(4)–C(3)	109.6 (6)
		O(4)–C(4)–C(5)	112.0 (6)
		O(5)–C(5)–C(4)	109.6 (6)
		O(5)–C(5)–C(6)	110.2 (6)
		O(6)–C(6)–C(5)	113.5 (6)

Table 3. Hydrogen-bond distances and angles

	Cell	Asymmetric unit of the second atom	Distance (\AA)	Angle ($^\circ$) O–H...O
Compound (1)				
O(1)···O(6)	120	2	2.969 (9)	160 (4)
O(2)···O(3)	11 $\bar{1}$	2	2.965 (9)	140 (4)
O(2)···O(4)	11 $\bar{1}$	2	2.819 (8)	123 (4)
O(2)···O(4)	110	2	2.911 (9)	157 (4)
O(3)···O(2)	110	2	2.965 (9)	140 (4)
O(4)···O(2)	11 $\bar{1}$	2	2.911 (9)	157 (4)
O(4)···O(2)	110	2	2.819 (9)	123 (4)
O(5)···O(3)	12 $\bar{1}$	2	2.778 (9)	150 (4)
Compound (2)				
O(1)···O(5)	001	4	2.637 (7)	161 (2)
O(2)···O(6)	0 $\bar{1}$ 1	4	2.901 (8)	138 (3)
O(3)···O(4)	1 $\bar{1}$ 1	4	2.769 (7)	144 (3)
O(3)···O(5)	0 $\bar{1}$ 1	4	2.720 (7)	129 (2)
O(4)···O(3)	101	4	2.769 (7)	144 (3)
O(4)···O(6)	1 $\bar{1}$ 1	4	2.819 (8)	150 (3)
O(5)···O(1)	011	4	2.637 (8)	161 (2)
O(5)···O(3)	001	4	2.720 (7)	129 (2)
O(6)···O(2)	001	4	2.901 (8)	138 (3)
O(6)···O(4)	101	4	2.819 (7)	150 (3)

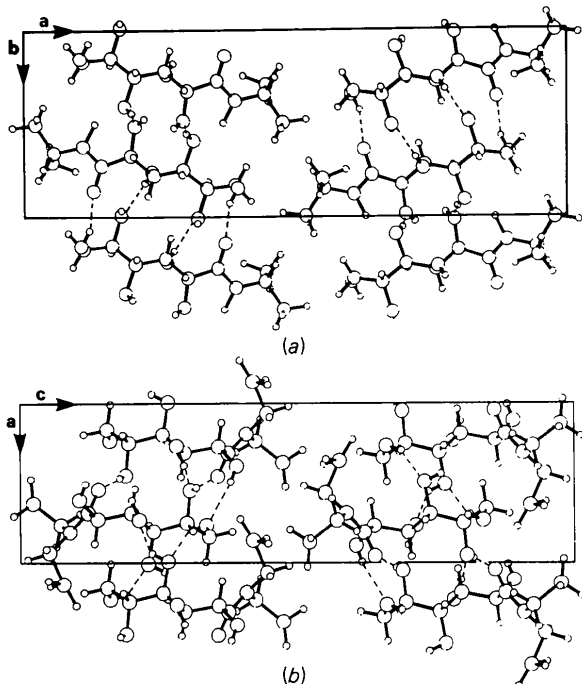


Fig. 2. The arrangement of the molecules in the unit cell (a) viewed along [001] for (1) and (b) along [010] for (2) with hydrogen bonds indicated by dotted lines.

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, ω - 2θ 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.