stable isomer that involved changing four-membered rings to five- and three-membered rings has been observed in two photodimers obtained from tyramine (Iwakuma, Nakai, Yonemitsu, Jones, Karle & Witkop, 1972).

Torsional angles are shown in Table 2. The five-membered ring containing a planar C(1) atom and a planar N(2) atom is approximately planar with maximum deviations of ±0·10 Å from the least-squares plane. The five-membered ring containing all saturated C atoms has the usual envelope conformation with atoms C(8), C(4), C(5) and C(6) nearly in a plane, with maximum deviations of ±0·05 Å from a least-squares plane and atom C(7) placed at 0·53 Å from that plane.

Pairs of hydrogen bonds between the OH and carbonyl O atoms link pairs of molecules around a center of symmetry. In the O(2)H···O(1) hydrogen bond, the O(2)—O(1) length is 2·751 Å. Another hydrogen bond, N(2)H···O(2), where N(2)—O(2) is 2·813 Å, links molecules head-to-tail along the **a** direction to complete a hydrogen-bonded layer, two molecules thick, in the **a** and **c** directions.

Table 2. *Torsional angles* (°)

E.s.d.'s are of the order of 0·6°.

C(8)C(1)N(2)N(3)	−13	C(5)C(6)C(7)C(8)	+30
C(1)N(2)N(3)C(4)	+2	C(6)C(7)C(8)C(4)	−36
N(2)N(3)C(4)C(8)	+10	C(7)C(8)C(4)C(5)	+29
N(3)C(4)C(8)C(1)	−17	N(3)C(4)C(5)C(9)	98
C(4)C(8)C(1)N(2)	+18	C(5)C(6)C(10)O(2)	177
C(8)C(4)C(5)C(6)	−10	C(6)C(10)O(2)H	61
C(4)C(5)C(6)C(7)	−13		

References

GILARDI, R. D. (1973). *Acta Cryst.* B**29**, 2089–2095.

HASEBE, M. (1980). PhD Thesis, Hokkaido Univ.

IWAKUMA, T., NAKAI, H., YONEMITSU, O., JONES, D. S., KARLE, I. L. & WITKOP, B. (1972). *J. Am. Chem. Soc.* **94**, 5136–5139.

KANAOKA, Y., HASEBE, M. & SATO, E. (1980). *Heterocycles*, **14**, 114.

KARLE, I. L. (1982). *Acta Cryst.* B**38**, 1019–1022.

Acta Cryst. (1982). B**38**, 1024–1027

Structure of 1-(*p*-Methoxyphenyl)-*trans*-1,2-diphenylbut-1-ene

BY S. D. CUTBUSH AND S. NEIDLE

Cancer Research Campaign Biomolecular Structure Research Group, Department of Biophysics, University of London King's College, 26–29 Drury Lane, London WC2B 5RL, England

A. B. FOSTER

Mass Spectrometry–Drug Metabolism Group, Institute of Cancer Research, Fulham Road, London SW3 6JB, England

AND F. LECLERCQ

Institut de Recherches Scientifiques sur le Cancer, CNRS, Boîte Postale n° 8, 94800 Villejuif, France

(*Received* 28 *July* 1981; *accepted* 16 *October* 1981)

Abstract. $C_{23}H_{22}O$, triclinic, $P\bar{1}$, $a = 9{\cdot}644$ (3), $b = 10{\cdot}0001$ (3), $c = 10{\cdot}4999$ (3) Å, $\alpha = 78{\cdot}77$ (3), $\beta = 67{\cdot}53$ (3), $\gamma = 74{\cdot}91$ (2)°, $V = 898{\cdot}3$ Å^3, $d_c = 1{\cdot}16$ Mg m^{-3}, $Z = 2$. There are two independent molecules in the asymmetric unit, which have very similar conformations. The structure was refined to a final R of 0·0586 for 2294 significant reflections.

Introduction. The title compound, 1-(*p*-methoxyphenyl)-*trans*-1,2-diphenylbut-1-ene, synthesized according to the method of Sohár, Ábráham, Schneider, Horváth & Fuggerth (1979), is a synthetic precursor of the antiœstrogenic drug tamoxifen {1-[*p*-(2-dimethylaminoethoxy)phenyl]-*trans*-1,2-diphenylbut-1-ene} which is in current use for the treatment of disseminated breast cancer.

A majority of breast tumours have identifiable œstrogen receptors, and their presence indicates a potential starting point for hormonal therapy (Camerman, Chan & Camerman, 1980). Œstradiol (the natural œstrogen) normally forms a complex with a protein receptor in the cytoplasm and this complex can then enter the nucleus and activate protein synthesis. Antiœstrogens compete with œstradiol for this receptor

0567-7408/82/031024-04$01.00

but the complex formed is incapable of promoting protein synthesis.

The *cis*-tamoxifen shows œstrogenic properties whereas the *trans* form exhibits weak œstrogenic and strong antiœstrogenic behaviour (Harper & Walpole, 1966). A comparison of the structures of œstradiol with *cis* and *trans* forms of tamoxifen (Weeks, Griffin & Duax, 1977) suggested that the presence of a bulky phenyl group *cis* in the functional phenyl group (as in the *trans* isomer) precludes œstrogenic activity but allows antiœstrogenic activity. It is possible that the *cis* isomers are similar enough to allow binding to the receptor but the position of the bulky phenyl group in the *trans* isomer does not allow reaction with nuclear DNA, which is a necessary prerequisite for protein synthesis.

Table 1. *Final fractional unit-cell coordinates for the non-*H *atoms* ($\times 10^4$) *and average thermal parameters* ($Å^2 \times 10^3$) *with e.s.d.'s in parentheses*

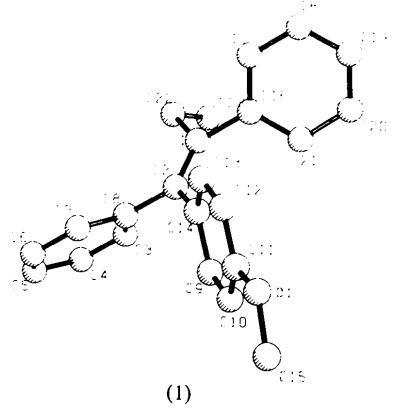
	x	y	z	$\langle U_{ii} \rangle$
(*a*) Molecule (1)				
C(1)	4597 (4)	−1453 (4)	1971 (4)	50 (2)
C(2)	3325 (4)	−0626 (4)	1794 (4)	48 (2)
C(3)	1958 (5)	−2300 (4)	1430 (5)	58 (2)
C(4)	1186 (5)	−2639 (5)	0723 (6)	69 (3)
C(5)	0889 (6)	−1770 (6)	−0386 (6)	79 (3)
C(6)	1388 (6)	−0534 (6)	−0751 (6)	79 (3)
C(7)	2144 (3)	−0198 (3)	−0044 (3)	41 (1)
C(8)	2504 (4)	−1057 (4)	1025 (4)	50 (2)
C(9)	1109 (4)	1142 (4)	3054 (4)	52 (2)
C(10)	0456 (5)	2467 (4)	3474 (4)	50 (2)
C(11)	1339 (4)	3462 (4)	3070 (4)	52 (2)
C(12)	2840 (5)	3145 (4)	2285 (5)	58 (3)
C(13)	3550 (5)	1815 (4)	1834 (4)	52 (2)
C(14)	2687 (4)	0763 (4)	2257 (4)	45 (2)
C(15)	−0778 (9)	5253 (7)	4134 (9)	100 (5)
C(16)	5406 (4)	−1071 (4)	2786 (4)	49 (2)
C(17)	6906 (5)	−0947 (5)	2185 (5)	64 (3)
C(18)	7677 (6)	−0635 (6)	2931 (7)	83 (3)
C(19)	6971 (7)	−0452 (6)	4290 (7)	97 (4)
C(20)	5507 (7)	−0607 (6)	4904 (5)	81 (4)
C(21)	4684 (5)	−0877 (5)	4161 (5)	66 (3)
C(22)	5354 (6)	−2839 (5)	1370 (6)	72 (3)
C(23)	5309 (9)	−4076 (6)	2467 (10)	92 (5)
O(1)	0795 (4)	4797 (3)	3434 (4)	78 (2)
(*b*) Molecule (2)				
C(1)	−4503 (4)	3756 (4)	8101 (4)	47 (2)
C(2)	−3209 (4)	2960 (3)	8279 (4)	45 (2)
C(3)	−2147 (9)	2472 (7)	10136 (7)	99 (4)
C(4)	−1267 (5)	2855 (5)	10817 (5)	70 (3)
C(5)	−0778 (5)	4075 (5)	10433 (5)	71 (3)
C(6)	−1021 (5)	4966 (4)	9324 (4)	65 (2)
C(7)	−1823 (4)	4620 (4)	8615 (4)	54 (2)
C(8)	−2351 (4)	3384 (4)	8990 (4)	46 (2)
C(9)	−1008 (4)	1151 (4)	7007 (4)	47 (2)
C(10)	−0326 (4)	−0159 (4)	6586 (4)	51 (2)
C(11)	−1186 (5)	−1165 (4)	6969 (4)	52 (2)
C(12)	−2752 (5)	−0837 (4)	7805 (4)	57 (2)
C(13)	−3384 (4)	0489 (4)	8164 (4)	51 (2)
C(14)	−2533 (4)	1501 (3)	7811 (3)	46 (2)
C(15)	0947 (6)	−2861 (5)	5782 (6)	70 (3)
C(16)	−5276 (4)	3366 (4)	7293 (4)	50 (2)
C(17)	−4531 (5)	3178 (4)	5874 (4)	56 (2)
C(18)	−5283 (7)	2886 (5)	5110 (5)	78 (3)
C(19)	−6849 (6)	2787 (5)	5793 (5)	76 (3)
C(20)	−7610 (5)	2970 (5)	7179 (5)	72 (2)
C(21)	−6830 (5)	3265 (4)	7922 (5)	59 (2)
C(22)	−5285 (5)	5146 (4)	8666 (4)	54 (2)
C(23)	−5132 (6)	6368 (4)	7545 (6)	71 (3)
O(1)	−0636 (3)	−2508 (3)	6622 (3)	68 (2)

Chunky crystals (0·4 × 0·2 × 0·1 mm) were obtained from ethanol. Accurate cell dimensions were obtained from 25 θ values measured on an Enraf–Nonius CAD-4 diffractometer.

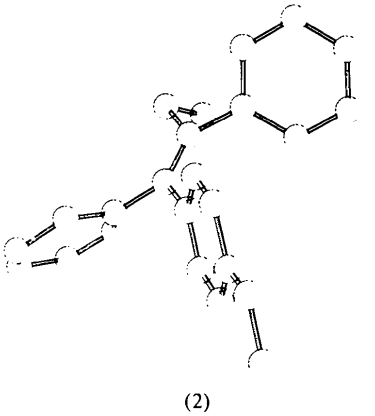
Intensities of 3247 unique reflections were measured on the diffractometer with graphite-monochromated Cu $K\alpha$ radiation ($1{\cdot}5 < \theta < 65{\cdot}0°$) using an ω–2θ scan technique. Periodic intensity checks on the selected reflections showed no decay therefore no correction was necessary.

A total of 2294 reflections were considered to have significant intensities [$F_o > 4\sigma(F_o)$] and were used in the subsequent analysis.

The structure was solved by direct methods, and refined by full-matrix least-squares techniques to a final reliability index R of 0·0586 and a weighted R_w of 0·0758. The positions of all the H atoms [except those attached to C(19) and C(20)] were found in a



(1)



(2)

Fig. 1. Molecules (1) and (2), viewed from an identical projection.

difference Fourier synthesis. The latter were calculated from geometric considerations. The H-atom parameters were kept fixed during refinement. Table 1 details the final non-H atomic coordinates.*

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36480 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

	Molecule (1)	Molecule (2)
C(1)–C(2)	1·346 (5)	1·352 (5)
C(2)–C(8)	1·505 (7)	1·484 (6)
C(3)–C(8)	1·402 (6)	1·407 (8)
C(3)–C(4)	1·368 (9)	1·456 (12)
C(4)–C(5)	1·387 (8)	1·355 (7)
C(5)–C(6)	1·376 (8)	1·379 (7)
C(6)–C(7)	1·353 (9)	1·397 (8)
C(7)–C(8)	1·374 (5)	1·388 (6)
C(2)–C(14)	1·461 (5)	1·529 (5)
C(9)–C(14)	1·417 (5)	1·376 (4)
C(9)–C(10)	1·388 (6)	1·378 (5)
C(10)–C(11)	1·376 (6)	1·373 (6)
C(11)–C(12)	1·352 (5)	1·414 (5)
C(12)–C(13)	1·409 (6)	1·368 (5)
C(13)–C(14)	1·410 (6)	1·375 (6)
C(11)–O(1)	1·373 (5)	1·375 (4)
O(1)–C(15)	1·403 (8)	1·430 (6)
C(1)–C(16)	1·510 (7)	1·480 (7)
C(16)–C(17)	1·368 (6)	1·411 (5)
C(17)–C(18)	1·387 (10)	1·379 (9)
C(18)–C(19)	1·352 (9)	1·423 (8)
C(19)–C(20)	1·346 (9)	1·381 (7)
C(20)–C(21)	1·406 (10)	1·385 (9)
C(16)–C(21)	1·369 (6)	1·410 (5)
C(1)–C(22)	1·521 (6)	1·512 (5)
C(22)–C(23)	1·514 (9)	1·517 (6)

Discussion. Fig. 1 shows the two independent molecules in the asymmetric unit, and Tables 2 and 3 detail their bond lengths and angles. In terms of these

Table 3. *Bond angles* (°) *with e.s.d.'s in parentheses*

	Molecule (1)	Molecule (2)
C(16)–C(1)–C(2)	122·4 (4)	123·0 (3)
C(16)–C(1)–C(22)	114·1 (4)	113·1 (3)
C(2)–C(1)–C(22)	123·6 (5)	123·9 (4)
C(1)–C(2)–C(14)	123·4 (4)	122·5 (4)
C(1)–C(2)–C(8)	121·3 (4)	123·8 (3)
C(8)–C(2)–C(14)	115·3 (3)	113·7 (3)
C(2)–C(8)–C(3)	122·0 (4)	115·6 (5)
C(2)–C(8)–C(7)	121·4 (4)	123·6 (3)
C(3)–C(8)–C(7)	116·4 (5)	120·7 (5)
C(4)–C(3)–C(8)	120·2 (4)	116·1 (6)
C(3)–C(4)–C(5)	122·0 (5)	121·6 (5)
C(4)–C(5)–C(6)	117·5 (6)	121·0 (6)
C(5)–C(6)–C(7)	120·4 (5)	119·2 (4)
C(6)–C(7)–C(8)	123·5 (4)	121·2 (4)
C(2)–C(14)–C(13)	122·4 (3)	122·8 (3)
C(2)–C(14)–C(9)	120·2 (4)	120·0 (3)
C(9)–C(14)–C(13)	117·1 (3)	117·2 (3)
C(14)–C(9)–C(10)	121·7 (4)	122·6 (4)
C(9)–C(10)–C(11)	119·7 (3)	119·4 (3)
C(10)–C(11)–C(12)	120·2 (4)	119·1 (3)
C(10)–C(11)–O(1)	123·9 (3)	124·9 (3)
O(1)–C(11)–C(12)	115·9 (4)	116·0 (4)
C(11)–O(1)–C(15)	119·6 (5)	116·8 (4)
C(11)–C(12)–C(13)	122·0 (4)	119·2 (4)
C(12)–C(13)–C(14)	119·2 (3)	122·4 (3)
C(1)–C(16)–C(17)	121·0 (4)	121·8 (3)
C(1)–C(16)–C(21)	121·4 (4)	119·7 (3)
C(17)–C(16)–C(21)	117·5 (5)	118·5 (5)
C(16)–C(17)–C(18)	121·6 (5)	121·5 (4)
C(17)–C(18)–C(19)	121·0 (5)	118·0 (5)
C(18)–C(19)–C(20)	118·0 (7)	121·9 (6)
C(19)–C(20)–C(21)	122·1 (5)	119·0 (5)
C(20)–C(21)–C(16)	119·6 (4)	121·2 (4)
C(1)–C(22)–C(23)	113·3 (5)	113·0 (3)

Table 4. *Comparison of dihedral and torsion angles in various tamoxifen structures*

Atoms defining the planes

Plane (1): C(3), C(4), C(5), C(6), C(7), C(8)
Plane (2): C(9), C(10), C(11), C(12), C(13), C(14)
Plane (3): C(16), C(17), C(18), C(19), C(20), C(21)
Plane (4): C(16), C(1), C(22), C(2), C(14), C(8)

Dihedral angles (°)

	This compound				(*c*)		
	Mol. (1)	Mol. (2)	(*a*)	(*b*)	Mol. (1)	Mol. (2)	(*d*)
Planes 1/4	54·3	55·7	64·2	55·1	55	53	–
Planes 2/4	55·7	54·5	50·4	55·0	52	60	49
Planes 3/4	61·9	61·2	56·9	57·5	55	49	56

Torsion angles (°)

	This compound				(*c*)		
	Mol. (1)	Mol. (2)	(*a*)	(*b*)	Mol. (1)	Mol. (2)	(*d*)
C(2)–C(1)–C(22)–C(23)	−115·0	109·3	114·0	–	−116	118	–
C(15)–O(1)–C(11)–C(10)	−7·0	−1·0	0·3	1·6	19	−14	−1·6
C(15)–O(1)–C(11)–C(12)	173·9	−179·7	179·9	−179·6	−166	167	−179·1

E.s.d.'s for dihedral and torsion angles in the present analysis are in the range 0·5 to 0·8°.

(*a*) Tamoxifen (Précigoux *et al.*, 1979). (*b*) Clomiphene (Ernst & Hite, 1976). (*c*) *cis*-Tamoxifen (Kilbourn & Owston, 1970). (*d*) Nafoxidine (Camerman *et al.*, 1980).

parameters, the two molecules are identical. However, Table 4 shows that there are small, though significant differences between the conformations of the two molecules, especially at the more flexible regions of the structures.

The molecules of the title compound adopt the propeller conformation seen in related structures: in tamoxifen (Précigoux, Courseille, Geoffre & Hospital, 1979), in its *cis* isomer (Kilbourn & Owston, 1970), in the 1-*p*-(diethylaminoethoxy) analogue clomiphene (Ernst & Hite, 1976), and in the 1-*p*-(2-pyrrolidenethoxy) derivative nafoxidine (Camerman *et al.*, 1980). In all cases, the angle between the phenyl rings is 50–60°. Each phenyl ring is planar within experimental error.

The present analysis has confirmed the *trans* arrangement of the ethyl group relative to the methoxyphenyl ring. This arrangement appears to be necessary for antiœstrogenic activity, and is found in the other active analogues investigated crystallographically (Camerman *et al.*, 1980). The high degree of conformational similarity noted by these authors, and shown in Table 4, does suggest a unique and specific protein receptor site for these molecules.

We are grateful to the Cancer Research Campaign (grant SP1384 and Career Development Award to SN), the Medical Research Council (to ABF), and EORTC (to FL) for support.

References

CAMERMAN, N., CHAN, L. Y. Y. & CAMERMAN, A. (1980). *J. Med. Chem.* **23**, 941–945.

ERNST, S. & HITE, G. (1976). *Acta Cryst.* B**32**, 291–293.

HARPER, M. J. K. & WALPOLE, A. L. (1966). *Nature (London)*, **212**, 87.

KILBOURN, B. T. & OWSTON, P. G. (1970). *J. Chem. Soc. B*, 1–5.

PRÉCIGOUX, G., COURSEILLE, C., GEOFFRE, S. & HOSPITAL, M. (1979). *Acta Cryst.* B**35**, 3070–3072.

SOHÁR, P., ÁBRÁHAM, G., SCHNEIDER, G., HORVÁTH, T. & FUGGERTH, E. (1979). *Acta Chim. Acad. Sci. Hung.* **100**, 69–74.

WEEKS, C. M., GRIFFIN, J. F. & DUAX, W. L. (1977). *Am. Crystallogr. Assoc. Summer Meet.*, Abstract PB6.

Acta Cryst. (1982). B**38**, 1027–1030

Amino-1 Désoxy-1 (Thioxométhylène)-*N*,*O*-1,2 β-D-Lyxopyrannose

PAR P. BRIARD ET R. ROQUES

Laboratoire de Cristallographie de l'Université Nationale de Côte d'Ivoire, 04 *BP* 322, *Abidjan* 04, *Côte d'Ivoire*

G. GOSSELIN ET J. L. IMBACH

Laboratoire de Chimie Bio-Organique, ERA n° 195 *du CNRS, Université des Sciences et Techniques du Languedoc,* 34060 *Montpellier CEDEX, France*

J. L. MONTERO

Laboratoire de Chimie Organique Physique et Thérapeutique, Université Nationale de Côte d'Ivoire, 04 *BP* 322 *Abidjan* 04, *Côte d'Ivoire*

ET J. P. DECLERCQ ET G. GERMAIN

Laboratoire de Chimie Physique et Cristallographie, Université de Louvain, 1348 *Louvain-la-Neuve, Belgique*

(*Reçu le* 1 *septembre* 1981, *accepté le* 20 *octobre* 1981)

Abstract. $C_6H_9NO_4S$, orthorhombic, space group $P2_12_12_1$, $a = 10{\cdot}588\,(2)$, $b = 7{\cdot}728\,(3)$, $c = 9{\cdot}693\,(2)$ Å, $Z = 4$, $V = 829{\cdot}4$ Å^3, $D_x = 1{\cdot}53$ Mg m^{-3}. The crystal structure has been determined from three-dimensional data collected with Cu $K\alpha$ radiation. Direct methods and least-squares refinements were employed. The structure was refined to $R = 0{\cdot}069$ for 651 observed reflexions. The results show that this compound has a pyranic cyclic structure and a β-[1,2] substitution.

Introduction. La réaction d'un ose avec le thiocyanate de potassium en solution aqueuse concentrée et en présence d'acide chlorhydrique, conduit aux dérivés thioxooxazolidiniques correspondants: c'est le cas pour

0567-7408/82/031027-04$01.00