O(3′)—N(5′)	1.26 (1)	N(4′)—C(6′)	1.36 (1)
O(4)—N(5)	1.24 (1)	C(1)—C(2)	1.49 (1)
O(4′)—N(5′)	1.23 (1)	C(1′)—C(2′)	1.49 (1)
N(1)—C(2)	1.48 (1)	C(2)—C(3)	1.53 (1)
N(1′)—C(2′)	1.50 (1)	C(2′)—C(3′)	1.53 (1)
N(2)—C(5)	1.47 (1)	C(3)—C(4)	1.33 (2)
N(2)—C(6)	1.33 (1)	C(3′)—C(4′)	1.48 (1)
N(2′)—C(5′)	1.48 (1)	C(4)—C(5)	1.44 (1)
N(2′)—C(6′)	1.32 (1)	C(4′)—C(5′)	1.53 (1)
C(5)—N(2)—C(6)	126.6 (9)	N(1)—C(2)—C(3)	111.9 (7)
C(5′)—N(2′)—C(6′)	126.5 (8)	C(1)—C(2)—C(3)	109 (1)
N(5)—N(4)—C(6)	118.8 (9)	N(1′)—C(2′)—C(1′)	107.6 (7)
N(5′)—N(4′)—C(6′)	118.5 (8)	N(1′)—C(2′)—C(3′)	111.3 (7)
O(3)—N(5)—O(4)	121.7 (8)	C(1′)—C(2′)—C(3′)	113.4 (8)
O(3)—N(5)—N(4)	113 (1)	C(2)—C(3)—C(4)	122 (1)
O(4)—N(5)—N(4)	125 (1)	C(2′)—C(3′)—C(4′)	115.4 (7)
O(3′)—N(5′)—O(4′)	121.0 (8)	C(3)—C(4)—C(5)	124 (2)
O(3′)—N(5′)—N(4′)	112.7 (9)	C(3′)—C(4′)—C(5′)	108.7 (8)
O(4′)—N(5′)—N(4′)	126.1 (9)	N(2)—C(5)—C(4)	110 (1)
O(1)—C(1)—O(2)	124.4 (9)	N(2′)—C(5′)—C(4′)	114.4 (8)
O(1)—C(1)—C(2)	112.4 (8)	N(2)—C(6)—N(3)	121 (1)
O(2)—C(1)—C(2)	123.2 (8)	N(2)—C(6)—N(4)	126.3 (9)
O(1′)—C(1′)—O(2′)	125 (1)	N(3)—C(6)—N(4)	113 (1)
O(1′)—C(1′)—C(2′)	113.2 (9)	N(2′)—C(6′)—N(3′)	120.6 (9)
O(2′)—C(1′)—C(2′)	122.1 (9)	N(2′)—C(6′)—N(4′)	127.1 (9)
N(1)—C(2)—C(1)	108.6 (7)	N(3′)—C(6′)—N(4′)	112.2 (9)

The crystal was sealed in a capillary to prevent sublimation. The positions of the H atoms on atoms C(3) and C(4) were calculated geometrically. Other H atoms were subsequently located in successive difference Fourier maps. Program used for data collection and cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Programs used to solve structure: *SHELXS*86 (Sheldrick, 1985) and *DIRDIF* (Beurskens, 1984). All calculations, including data reduction, were carried out using the *TEXSAN* crystallographic package (Molecular Structure Corporation, 1985).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, torsion angles and hydrogen-bond geometry details have been deposited with the IUCr (Reference: AS1098). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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trans-1-(4-Iodophenyl)-2-phenyl-1-{4-[2-(1-pyrrolidinyl)ethoxy]phenyl}-1-butene, $C_{28}H_{30}INO$

CHRISTINE M. NUNN AND ESTHER J. PARKER

The CRC Biomolecular Structure Unit, Institute of Cancer Research, 15 Cotswold Road, Sutton, Surrey SM2 5NG, England

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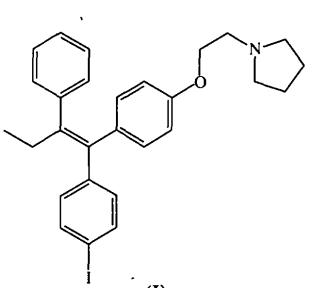
Abstract

The title compound is a tamoxifen derivative. The crystal structure shows a central ethylene bond about which three phenyl rings adopt a propeller conformation. The [2-(1-pyrrolidinyl)ethoxy]phenyl ring lies *trans* with respect to the ethyl group across the ethylene bond.

Comment

This study reports the structure of a *trans*-tamoxifen derivative. *trans*-Tamoxifen and its derivatives show antioestrogenic activity and are important for the treatment of hormone-sensitive breast cancer. A number of tamoxifen structures have been reported previously, and include *trans*-tamoxifen (Precigoux, Courseille, Geoffre & Hospital, 1979), 2-hydroxytamoxifen, 3-hydroxytamoxifen, 2-methyl-4-hydroxytamoxifen (Kuroda, Cutbush, Neidle & Leung, 1985), 4-methylthiotamoxifen (Blackburn, Goodman & Smith, 1988) and tamoxifen citrate (Goldberg & Becker, 1987). The structure of the *E* and *Z* isomers of an iodotamoxifen have been reported (Hunter, Payne, Rahman, Richardson & Ponce, 1983). The *Z* isomer of iodotamoxifen differs from the title compound (I) in having a dimethylammine group in place of the pyrrolidinyl group and a different iodo-substituted phenyl ring.



(I)

The overall conformation of the structure is similar to that seen for the other tamoxifen structures. The central triphenylethylene system adopts a propeller conformation. The dihedral angles formed between the (1) unsubstituted phenyl, (2) 4-iodophenyl and (3) [2-

(1-pyrrolidinyl)ethoxy]phenyl planes are (1)^(2) 56.3, (1)^(3) 58.6 and (2)^(3) 102.1°. The phenyl rings are not coplanar with the ethylene bond. The dihedral angle between the planes C1,C11,C17 and C2,C3,C9 is 12.1°, with angles C11—C1—C17 and C3—C2—C9 of 113.8 (2) and 114.9 (2)°, respectively, smaller than the expected value of 120°. There is no difference between the C—I and C=C bond lengths seen in this structure and those found for iodotamoxifen.

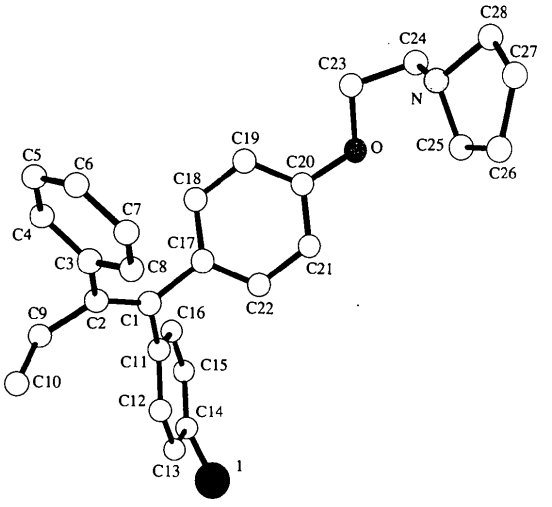


Fig. 1. View of the molecule and the numbering scheme for all non-H atoms.

Experimental

Crystals of the title compound were grown from a 95% methanol solution by slow evaporation at room temperature.

Crystal data

$C_{28}H_{30}INO$	Mo $K\alpha$ radiation
M_r = 523.46	λ = 0.71069 Å
Monoclinic	Cell parameters from 10 reflections
$C2/c$	θ = 13–14°
a = 32.649 (8) Å	μ = 1.31 mm^{-1}
b = 6.1165 (7) Å	T = 293 K
c = 29.739 (3) Å	Rectangular prism
β = 124.36 (1)°	0.6 × 0.3 × 0.3 mm
V = 4902 (1) Å^3	Colourless
Z = 8	
D_x = 1.42 Mg m^{-3}	

Data collection

Enraf–Nonius CAD-4 diffractometer	3080 observed reflections [$I > 2\sigma(I)$]
$\omega/2\theta$ scans	θ_{max} = 22°
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)	$h = 0 \rightarrow 35$
	$k = 0 \rightarrow 10$
T_{min} = 0.91, T_{max} = 1.15	$l = -35 \rightarrow 35$
4053 measured reflections	2 standard reflections
4053 independent reflections	frequency: 107 min
	intensity variation: 1%

Refinement

Refinement on F	$(\Delta/\sigma)_{max}$ = 0.01
R = 0.028	$\Delta\rho_{max}$ = 0.30 e Å^{-3}
wR = 0.041	$\Delta\rho_{min}$ = −0.21 e Å^{-3}
S = 1.63	Extinction correction: none
2647 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
280 parameters	
Only coordinates of H atoms refined	
$w = 1/\sigma^2(F)$	

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
I	0.49427 (1)	0.40181 (4)	0.15983 (1)	4.943 (7)
O	0.43789 (8)	1.1303 (4)	0.40957 (8)	4.61 (6)
N	0.4082 (1)	1.1384 (5)	0.4882 (1)	4.68 (8)
C1	0.34727 (9)	0.9948 (6)	0.1842 (1)	3.29 (7)
C2	0.3016 (1)	1.0621 (5)	0.1442 (1)	3.60 (8)
C3	0.2661 (1)	1.1507 (6)	0.1558 (1)	3.48 (8)
C4	0.2431 (1)	1.3522 (6)	0.1365 (2)	5.1 (1)
C5	0.2094 (1)	1.4284 (7)	0.1475 (2)	6.3 (1)
C6	0.1980 (1)	1.3061 (9)	0.1765 (2)	6.6 (1)
C7	0.2196 (1)	1.1022 (7)	0.1958 (1)	6.0 (1)
C8	0.2533 (1)	1.0289 (7)	0.1852 (1)	4.33 (9)
C9	0.2821 (1)	1.0511 (7)	0.0844 (1)	5.2 (1)
C10	0.2377 (2)	0.9113 (8)	0.0501 (2)	6.9 (1)
C11	0.37919 (9)	0.8645 (5)	0.1728 (1)	3.18 (7)
C12	0.3652 (1)	0.6560 (6)	0.1498 (1)	4.02 (8)
C13	0.3967 (1)	0.5262 (6)	0.1446 (1)	4.22 (9)
C14	0.4431 (1)	0.6039 (5)	0.1620 (1)	3.47 (8)
C15	0.4574 (1)	0.8096 (5)	0.1828 (1)	3.46 (7)
C16	0.4259 (1)	0.9379 (5)	0.1891 (1)	3.40 (7)
C17	0.37066 (9)	1.0386 (5)	0.2432 (1)	2.99 (7)
C18	0.4004 (1)	0.8796 (5)	0.2823 (1)	3.51 (8)
C19	0.4223 (1)	0.9149 (5)	0.3367 (1)	3.59 (8)
C20	0.4153 (1)	1.1145 (5)	0.3545 (1)	3.36 (7)
C21	0.38813 (9)	1.2756 (5)	0.3173 (1)	3.45 (7)
C22	0.36610 (9)	1.2358 (5)	0.2624 (1)	3.36 (8)
C23	0.4294 (1)	1.3203 (6)	0.4308 (1)	5.01 (9)
C24	0.4442 (1)	1.2713 (7)	0.4876 (1)	5.3 (1)
C25	0.4129 (2)	0.9067 (7)	0.4835 (2)	6.6 (1)
C26	0.3815 (2)	0.8043 (9)	0.5012 (2)	8.0 (1)
C27	0.3763 (2)	0.9775 (9)	0.5328 (2)	8.1 (1)
C28	0.4095 (2)	1.1584 (8)	0.5377 (1)	7.5 (1)

Table 2. *Selected geometric parameters* (Å, °)

I—C14	2.107 (4)	C17—C22	1.378 (5)
C9—C10	1.484 (5)	C2—C3	1.488 (6)
O—C20	1.367 (4)	C18—C19	1.365 (4)
C11—C12	1.396 (5)	C2—C9	1.515 (5)
O—C23	1.423 (5)	C19—C20	1.401 (5)
C11—C16	1.388 (4)	C3—C4	1.387 (5)
N—C24	1.440 (6)	C20—C21	1.369 (4)
C12—C13	1.376 (6)	C3—C8	1.377 (6)
N—C25	1.441 (5)	C21—C22	1.386 (4)
C13—C14	1.380 (5)	C4—C5	1.392 (7)
N—C28	1.453 (6)	C23—C24	1.500 (5)
C14—C15	1.363 (4)	C5—C6	1.344 (8)
C1—C2	1.342 (3)	C25—C26	1.525 (9)
C15—C16	1.386 (5)	C6—C7	1.388 (6)
C1—C11	1.495 (5)	C26—C27	1.487 (8)
C17—C18	1.403 (4)	C7—C8	1.380 (7)
C1—C17	1.491 (4)	C27—C28	1.497 (8)
C20—O—C23	118.7 (2)	C18—C19—C20	119.9 (3)
C11—C12—C13	121.5 (3)	C4—C3—C8	117.2 (4)
C24—N—C25	114.9 (4)	O—C20—C19	115.0 (2)
C12—C13—C14	119.5 (3)	C3—C4—C5	120.9 (4)

C24—N—C28	114.1 (3)	O—C20—C21	125.6 (3)
I—C14—C13	120.5 (2)	C4—C5—C6	120.4 (4)
C25—N—C28	103.7 (4)	C19—C20—C21	119.4 (3)
I—C14—C15	118.9 (2)	C5—C6—C7	120.3 (5)
C2—C1—C11	122.1 (3)	C20—C21—C22	119.7 (3)
C13—C14—C15	120.5 (3)	C6—C7—C8	118.9 (4)
C2—C1—C17	124.1 (3)	C17—C22—C21	122.5 (3)
C14—C15—C16	119.7 (3)	C3—C8—C7	122.2 (4)
C11—C1—C17	113.8 (2)	O—C23—C24	108.4 (3)
C11—C16—C15	121.4 (3)	C2—C9—C10	115.7 (4)
C1—C2—C3	121.9 (3)	N—C24—C23	112.3 (2)
C1—C17—C18	120.5 (3)	C1—C11—C12	121.4 (3)
C1—C2—C9	123.1 (4)	N—C25—C26	104.0 (4)
C1—C17—C22	122.8 (3)	C1—C11—C16	121.1 (3)
C3—C2—C9	114.9 (2)	C25—C26—C27	105.3 (4)
C18—C17—C22	116.6 (3)	C12—C11—C16	117.3 (3)
C2—C3—C4	122.5 (4)	C26—C27—C28	104.7 (5)
C17—C18—C19	121.9 (3)	N—C28—C27	103.4 (3)
C2—C3—C8	120.2 (3)		

The structure was solved using Patterson techniques. H atoms were placed at 0.96 Å from their associated C atom. They were refined in riding mode with a fixed thermal parameter of 1.2 times that of their associated C atom.

Data collection: *CAD*-4 (Enraf–Nonius, 1977). Cell refinement: *SETANG* (Enraf–Nonius, 1977). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *MolEN*. Program(s) used to refine structure: *MolEN*. Molecular graphics: *Ball & Stick* (Müller & Falk, 1987–1991).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: LI1116). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Deoxyuridine Derivative

MICHAEL BOLTE

Institut für Organische Chemie der Universität Frankfurt, Marie-Curie-Strasse 11, D-60439 Frankfurt am Main, Germany

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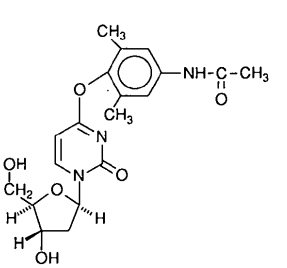
Abstract

The structure of *N*-(4-{1-[(2*R*,4*S*,5*R*)-4-hydroxy-5-(hydroxymethyl)-2-tetrahydrofuranyl]-2-oxo-1,2-dihydro-4-pyrimidinyloxy}-3,5-dimethylphenyl)acetamide chloroform solvate, $C_{19}H_{23}N_3O_6.CHCl_3$, has been determined and its geometrical features are described. This molecule plays an important role in the convertible nucleoside approach, a method of synthesizing oligodeoxynucleotides bearing tethered functionality.

Comment

Synthetic oligodeoxynucleotides are able to bind nucleic acid targets with high affinity and specificity. Unfortunately, they lack an ability to report binding or to effect chemical modification of the target sequence (MacMillan & Verdine, 1990). A method to attach non-natural ligands to synthetic DNA uses functionally tethered oligodeoxynucleotides (FTOs). When the tethered nucleophile is introduced at the monomer level of DNA synthesis the method is called 'nonconvergent'. Since this method bears some disadvantages, MacMillan & Verdine (1991) developed the 'convergent' strategy, where the tether is installed at the final step of DNA synthesis. This method leads to a variety of FTOs differing in sequence and tether structure starting from a single nucleoside precursor. The title compound, (I), is an aryl ether of uridine which can serve as a latent cytidine residue because it undergoes clean substitution when it is treated with ammonia (Zhou & Chattopadhyaya, 1986).



(I)