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Tropinyl 2-Isopropylbenzo[*b*]thiophene-3-carboxylate, $C_{20}H_{25}NO_2S$

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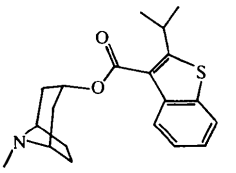
Abstract

The title tropinyl ester, 8-methyl-8-azabicyclo[3.2.1]oct-3-yl 2-isopropylbenzo[*b*]thiophene-3-carboxylate, is an analogue of the parasympathomimetic neurotransmitter acetyl choline. The molecule is stabilized in the observed conformation by an intramolecular C···O interaction.

Comment

Certain benzofused heterocycles, *viz.* benzo[*b*]thiophenes (Campaigne, Knapp, Neiss & Bosin, 1970; Bosin & Campaigne, 1977), substituted at various positions, have been found to selectively inhibit the action of thromboxane synthase without significantly inhibiting the action of prostacyclin synthase or cyclooxygenase. They are, therefore, useful as therapeutic agents for the treatment of thrombosis, ischaemic heart disease, stroke and migrane. We synthesized the title compound, (I), an analogue of the parasympathomimetic neurotransmitter acetyl choline, with the expectation that it may have a wide range of pharmacological effects.



(I)

The structure of (I) was confirmed by IR, ^{1}H NMR and mass spectroscopy, and elemental analyses. The three-dimensional structure of the tropine ester has now been determined by X-ray diffraction methods. An *ORTEP*II (Johnson, 1976) diagram with the atomic numbering scheme is shown in Fig. 1.

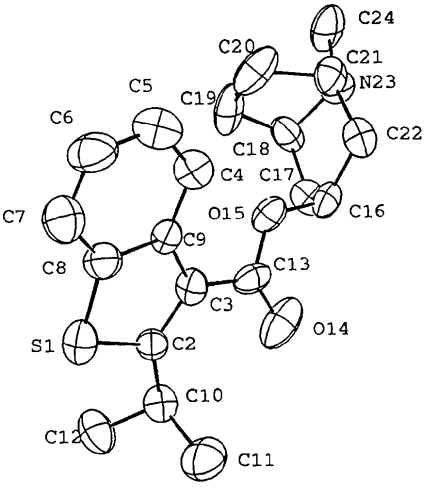


Fig. 1. *ORTEP*II (Johnson, 1976) plot of the molecule with displacement ellipsoids at the 50% probability level.

Beers & Reich (1970) have reported several partly and fully rigid molecules that are active as agonists or antagonists of acetyl choline. Conformational analysis of several of these agonists and antagonists can be used as a basis for the definition of structural parameters necessary for the range of activity of this class of compounds. A distance of about 5.9 Å between the receptor anionic site and the positively charged centre of the molecule plays a crucial role in their biological functions. The intramolecular N23···O15 distance (5.9 Å) in the present structure, which is similar to that observed in strychnine (Robertson & Beevers, 1951), indicates the suitability of the compound as a potential drug. The observed conformation of the molecule (Fig. 1) appears to be stabilized in part by a C—H···O interaction [C10···O14 = 2.96 (9) Å] and the restrictions introduced by the steric interactions between C12 and C11 on the one hand and O14 on the other. In any case, the N23···O15 distance is unaffected by the conformation of this part of the molecule.

Experimental

Esterification of 2-isopropylbenzo[*b*]thiophene-3-carboxylic acid chloride with tropine produced the title compound. The crystal density D_m was measured by flotation in benzene–chloroform mixture.

Crystal data

$C_{20}H_{25}NO_2S$
$M_r = 343.5$
Monoclinic
$P2_1/n$
$a = 13.887\ (2)$ Å
$b = 8.121\ (1)$ Å
$c = 16.201\ (3)$ Å
$\beta = 98.15\ (1)°$
$V = 1808.6\ (3)$ Å^3
$Z = 4$
$D_x = 1.26$ Mg m^{-3}
$D_m = 1.265$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 50 reflections
$\theta = 25–30°$
$\mu = 0.182$ mm^{-1}
$T = 300$ K
Needle
0.40 × 0.10 × 0.07 mm
White

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
3213 measured reflections
3213 independent reflections
2401 observed reflections [$I > 3\sigma(I)$]
$\theta_{max} = 25°$
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 9$
$l = -19 \rightarrow 19$
5 standard reflections monitored every 50 reflections
intensity decay: 1%

Refinement

Refinement on F
$R = 0.040$
$wR = 0.041$
$S = 1.01$
2401 reflections
317 parameters
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.38$
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *SHELXS86* (Sheldrick, 1985)

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

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
S	0.7615 (2)	0.2095 (3)	0.2772 (2)	0.060 (1)
C2	0.8663 (6)	0.1183 (10)	0.3252 (5)	0.046 (3)
C3	0.9168 (6)	0.0418 (10)	0.2686 (5)	0.042 (3)
C4	0.8994 (6)	0.0108 (10)	0.1066 (6)	0.051 (3)
C5	0.8428 (7)	0.0504 (12)	0.0339 (6)	0.063 (4)
C6	0.7585 (8)	0.1363 (14)	0.0330 (6)	0.075 (4)
C7	0.7271 (6)	0.1901 (13)	0.1033 (7)	0.073 (4)
C8	0.7840 (6)	0.1526 (11)	0.1795 (5)	0.053 (3)
C9	0.8711 (5)	0.0637 (9)	0.1824 (6)	0.039 (3)
C10	0.8891 (6)	0.1273 (11)	0.4200 (5)	0.051 (4)
C11	0.9534 (7)	0.2776 (13)	0.4439 (6)	0.083 (4)
C12	0.7984 (6)	0.1337 (12)	0.4616 (6)	0.070 (4)
C13	1.0094 (6)	−0.0463 (11)	0.2961 (6)	0.049 (3)
O14	1.0606 (5)	−0.0230 (9)	0.3604 (4)	0.083 (3)
O15	1.0285 (4)	−0.1547 (7)	0.2392 (3)	0.049 (2)
C16	1.1197 (6)	−0.2484 (11)	0.2562 (5)	0.049 (3)
C17	1.1094 (6)	−0.4026 (11)	0.3073 (5)	0.055 (4)
C18	1.0734 (6)	−0.5464 (11)	0.2524 (6)	0.059 (4)
C19	0.9734 (6)	−0.5119 (11)	0.1982 (8)	0.076 (4)
C20	0.9999 (7)	−0.4405 (12)	0.1185 (6)	0.074 (4)
C21	1.1118 (6)	−0.4387 (10)	0.1319 (5)	0.056 (4)
C22	1.1533 (6)	−0.2802 (12)	0.1730 (5)	0.053 (3)
N23	1.1398 (5)	−0.5741 (9)	0.1904 (4)	0.074 (4)
C24	1.1263 (7)	−0.7343 (12)	0.1499 (6)	0.050 (4)

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

S—C2	1.717 (8)	N23—C18	1.474 (12)
S—C8	1.719 (9)	N23—C21	1.468 (11)
O15—C13	1.328 (10)	N23—C24	1.457 (12)
O15—C16	1.469 (10)	C13—O14	1.190 (10)
C2—S—C8	92.7 (4)	C3—C2—C10	129.7 (7)
C13—O15—C16	117.7 (6)	N23—C21—C20	104.7 (7)
O15—C13—C3	111.1 (7)	C21—C20—C19	104.3 (7)
C3—C13—O14	124.8 (8)		

C9—C3—C13—O14	156.0 (9)
C9—C3—C2—C10	−179.3 (8)
C18—N23—C21—C22	74.5 (8)
C13—O15—C16—C17	86.2 (9)
C13—O15—C16—C22	−147.6 (7)
C16—O15—C13—C3	178.1 (6)
C21—C22—C16—O15	−88.7 (8)
C16—C22—C21—C20	57.7 (9)
C16—C22—C21—N23	−57.0 (9)
C2—C3—C13—O15	158.8 (8)
S—C2—C10—C11	−93.0 (8)
C3—C2—C10—C11	89.0 (10)
C24—N23—C21—C22	−165.0 (7)
C24—N23—C18—C17	164.5 (7)
C24—N23—C18—C19	−74.9 (9)
O15—C16—C17—C18	86.3 (8)
C3—C2—C10—C12	−147.9 (9)
C13—C3—C9—C8	179.7 (8)

The structure was solved using *SHELXS86* (Sheldrick, 1985) and successive difference Fourier syntheses. All H atoms except for the methyl H atoms were located from ΔF syntheses; methyl H atoms were calculated geometrically. All 24 non-H atoms were refined anisotropically and all H atoms isotropically using *SHELX76* (Sheldrick, 1976). The geometrical parameters of the molecule were calculated using *PARST* (Nardelli, 1983). All calculations were carried out on PC/AT 386 and MicroVAX II computers.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: VJ1009). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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4-Benzylbiphenyl

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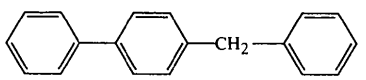
Abstract

In the title compound, $C_{19}H_{16}$, the average C—C bond length in all three phenyl rings is 1.380 (2) Å. Unlike in biphenyl itself, the magnitude of the torsion angle between the phenyl rings, C(2)—C(1)—C(7)—C(8), is −41.6 (4)°. The benzyl group is inclined towards the biphenyl moiety at a dihedral angle of 106.4 (4)°.

Comment

There is substantial literature on the studies of biphenyl and its derivatives. The work of Brock and co-workers (Brock & Haller, 1984; Brock & Morelan, 1986) on 4-hydroxybiphenyl shows that not only are some of these materials difficult to crystallize, but also that a given material may exist in more than one crystalline form. Their compound, like biphenyl itself, has the two phenyl rings coplanar in the solid state (Trotter, 1961; Hargreaves & Rizvi, 1962; Charbonneau & Delugeard, 1976; 1977), unlike in the gas phase (Almenningen & Bastiansen, 1958; Bastiansen & Traetteberg, 1962) where it displays a dihedral twist of 42°.



(I)

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The range of crystallization conditions reported so far indicates to us that biphenyl systems would be good subjects for a systematic analysis of growth conditions and morphology modifiers for the preparation of organic crystals from organic solvents. The first stage in this work concerns the crystallization of 4-substituted biphenyls from non-aqueous solutions. The present work reports the crystal and molecular structure of the title compound, (I). A perspective view of the molecule with atomic labelling is given in Fig. 1 and the unit-cell packing viewed down the *b* axis is presented in Fig. 2.

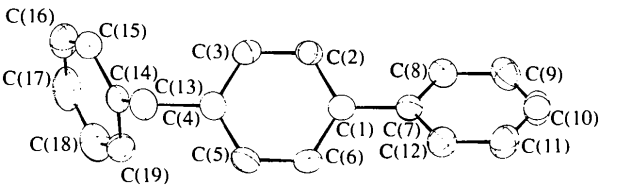

Fig. 1. A general view of 4-benzylbiphenyl. Displacement ellipsoids are plotted at the 50% probability level.

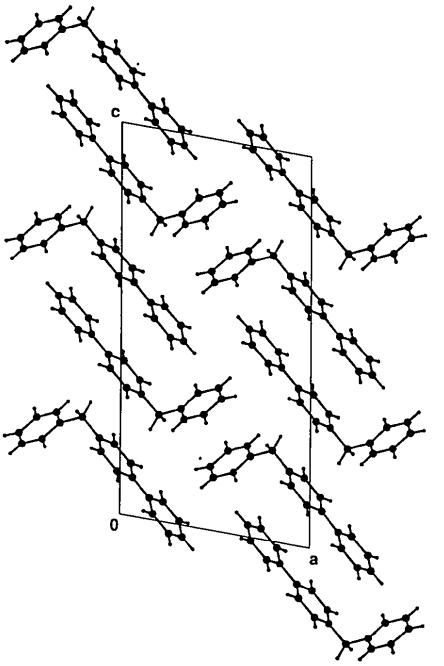

Fig. 2. Molecular packing of 4-benzylbiphenyl as seen down the *b* axis.

The average bond distances for the C(1)–C(6) and C(7)–C(12) phenyl rings are in good agreement with those of other 4-substituted biphenyls. The three internal ring bond angles, at C(1) [117.3 (2)°], C(4) [117.4 (2)°] and C(7) [117.5 (3)°], are significantly smaller than the ideal angle of 120°, but similar to those found in related compounds (Brock & Haller, 1984; Brock & Morelan, 1986). The length of the C(1)—C(7) bond [1.485 (4) Å] is quite close to the standard value for a single-bond length between trigonally linked C atoms