quality of data and many hydrogen positions, fixed geometrically, were not refined. The final (shift/e.s.d)$_{ave}$ = 0·033 and (shift/e.s.d.)$_{max}$ = 0·097. A final difference Fourier showed no peaks ≥0·3 e Å^{-3}. Scattering factors for non-hydrogen atoms were taken from *International Tables for X-ray Crystallography* (1968) and for hydrogens from Stewart, Davidson & Simpson (1965).

Discussion. The final fractional coordinates with equivalent temperature factors of non-hydrogens are given in Table 1. Thermal ellipsoids drawn at 50% probability level for non-hydrogens using *ORTEP* (Johnson, 1965) are shown in Fig. 1.*

The bond lengths and angles involving non-hydrogen atoms are shown in Figs. 2(*a*) and 2(*b*) respectively. The e.s.d.'s in bond lengths are 0·010 Å and in bond angles 0·5°. The bond lengths and angles in the two benzofuran ring systems are comparable with those in similar structures.

The torsion angles around the two furan rings *B* and *C* show that they are in envelope conformation with C_s = 1·39 and 0·80 respectively (Duax, Weeks & Rohrer, 1976). In both cases, the envelope atom C9 is out of the plane of the other four atoms in the furan rings *B* and *C* by 0·2 and 0·16 Å respectively. The two benzofuran moieties are planar. The angle between the planes of the two furan rings is 66·5°. This compares with 64·8° in a dihydrobenzofuro-[3,2-*b*]benzofuran derivative (Wong & Jurd, 1984).

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52300 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The two methyl carbons C1 and C16 are in the plane of the benzene rings to which they are attached. The torsion angle C17—C8—C9—C18, −13·9 (1)°, shows that C17 and C18 are *cis* with respect to the C8—C9 bond.

A stereoview of the unit-cell packing (Motherwell, 1978) is shown in Fig. 3. The molecules are stabilized in the unit cell by van der Waals interactions.

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Structure of the Thromboxane Receptor Antagonist EP 092

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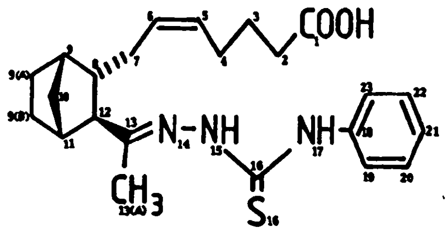
Abstract. 7-{3-[1-(4-Phenylthiosemicarbazono)ethyl]-bicyclo[2.2.1]hept-2-yl}-5-heptenoic acid, $C_{23}H_{31}N_3O_2S$, M_r = 413·7, monoclinic, $P2_1/a$, a = 12·9650 (7), b = 11·2081 (6), c = 16·8941 (12) Å, β = 110·452 (5)°, V = 2300·2 Å^3, Z = 4, D_x = 1·194 g cm^{-3}, Mo $K\alpha$, λ = 0·71073 Å, μ = 1·55 cm^{-1}, $F(000)$ = 888, T = 298 K.

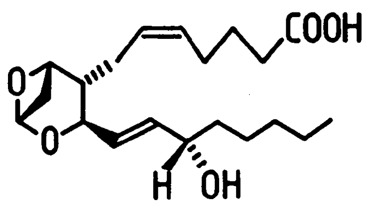
0108-2701/90/030447-04$03.00

Final $R = 0{\cdot}0472$ with 2778 independent data. EP 092 is a thromboxane receptor antagonist akin to many other analogues of the thromboxane A_2 [Wilson & Jones (1985). *Adv. Prostaglandin Thromboxane Leukotrine Res.* **14**, 393–425].

Introduction. EP 092, (±5)-*endo*-(6′-carboxyhex-2′*Z*-enyl)-6-*exo*-{1-[(*N*-phenylthiocarbamoyl)hydrazono]-ethyl}bicyclo[2.2.1]heptane, is a prostaglandin analogue. It acts as an antagonist to thromboxane A_2 (TXA_2) receptors (*i.e.* it is a ligand that binds tightly to the receptor, but fails to elicit an overt response thereby blocking the action of the natural agonist), which are involved in the control of vasoconstriction and platelet aggregation. The structure of EP 092 can be seen in the scheme where it is compared with the prostaglandin, thromboxane A_2.



EP 092



TXA_2

Experimental. Crystals were obtained as columns from ethanol at 243 K. A suitable crystal of dimensions $0{\cdot}5 \times 0{\cdot}45 \times 0{\cdot}9$ mm was selected and characterized by Weissenberg photography. The data set was collected on a Stoe STADI-4 diffractometer using graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71073$ Å). The orientation matrix used for data collection was derived from 11 reflections with $32 \leq 2\theta \leq 33°$, whilst accurate unit-cell dimensions were determined using 41 reflections measured at $+\omega$ in the same 2θ scan range. The range of indices collected was $-13 \leq h \leq 13$, $0 \leq k \leq 12$, $0 \leq l \leq 17$ and the standard reflections $\bar{8},1,11$, $\bar{5}69$ and 284 indicated no significant crystal movements or decay during the period of data collection.

A trial position for the S atom was found from a Patterson map, but a difference Fourier map calculated using this sulfur was uninterpretable. A direct-methods approach using *SHELXS*86 (Sheldrick, 1986) located all non-H atoms. These atoms were refined anisotropically before the addition of H atoms, which were added in their calculated positions and allowed to ride on the atoms to which they were attached, with fixed isotropic temperature factors and site occupancies. The maximum atom shift (Δ/σ) in the final run was 0·036. The standard *SHELX* weighting scheme was applied but proved to be no better than unit weights, which were therefore adopted.

During data collection 3198 reflections were measured, of which 2778 were used in refinement. 420 reflections with $I < 2\sigma$ were considered unobserved. The total number of parameters refined was 265 and the final R factor was 0·0472. The final difference map had maximum and minimum values of $+0{\cdot}21$ and $-0{\cdot}28$ e Å^{-3} respectively and the maximum $\sin\theta/\lambda$ was 0·538 Å^{-1}.

The program *CALC* (Gould & Taylor, 1983) was used to provide the molecular geometry data presented in Table 2. Diagrams were prepared using *PLUTO* (Motherwell, 1972) and an interactive version of *ORTEP* (Johnson, 1965; Mallinson & Muir, 1985). Atomic scattering factors were from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are given in Table 1.*

Discussion. The crystal structure of EP 092 is shown in Fig. 1. Table 2 lists bond lengths, bond angles and torsion angles. The bond lengths and torsion angles of EP 092 were compared with those of thromboxane B_2 (TXB_2; Fortier, Erman, Langs & DeTitta, 1980), for which a crystal structure exists. TXB_2 differs from TXA_2 in the addition of H_2O at the anomeric carbon which converts the acetal of TXA_2 into a hemiacetal. The torsion angle about C(4)—C(5) showed a difference of 164°. Superimposing the atoms C(7)—C(8)—C(12) shows a torsion angle change of 90° about the C(7)—C(8) bond. Further, the torsion angle about the C(12)—C(13) bond differs by 110° which superimposes the methyl group, C(13*A*), approximately upon the TXB_2 arm. These differences appear to result from the different ring structures, although the crystal packing may also have an effect. A comparison was also made between the bridged ring of the EP 092 and the two occurrences of the same bicyclo moiety in 1-bromo-3-[3-(2-methylenecyclohex-5-en-1-yl)bicyclo[2.2.1]-hept-2-yl]-4-bicyclo[2.2.1]hept-2-ylbenzene (Bocelli, Catellani & Chiusoli, 1984). No significant differences were found between these rigid structures. The

* Lists of structure factors and anisotropic thermal parameters have been deposited with ther British Library Document Supply Centre as Supplementary Publication No. SUP 52379 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates of atoms, with estimated standard deviations in parentheses*

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}(Å^2)
C(1)	0·93623 (25)	0·8608 (3)	−0·11108 (18)	0·0686 (21)
O(1)	0·96601 (20)	0·76023 (19)	−0·11369 (15)	0·0976 (19)
O(1*A*)	0·96605 (24)	0·94913 (21)	−0·14913 (18)	0·1181 (23)
C(2)	0·86217 (25)	0·9001 (3)	−0·06505 (19)	0·0730 (21)
C(3)	0·8319 (3)	0·8032 (3)	−0·01590 (19)	0·0754 (22)
C(4)	0·7564 (3)	0·8477 (3)	0·02980 (19)	0·0788 (23)
C(5)	0·71328 (25)	0·7483 (3)	0·06771 (21)	0·0778 (24)
C(6)	0·73448 (23)	0·7250 (3)	0·14805 (20)	0·0676 (21)
C(7)	0·80924 (22)	0·79234 (23)	0·22276 (17)	0·0616 (19)
C(8)	0·89218 (21)	0·70838 (22)	0·28517 (16)	0·0552 (17)
C(9)	0·95602 (25)	0·76060 (24)	0·37282 (17)	0·0679 (20)
C(9*A*)	1·0200 (3)	0·87260 (25)	0·36632 (19)	0·0724 (21)
C(9*B*)	1·1132 (3)	0·82250 (25)	0·33732 (20)	0·0738 (22)
C(10)	1·04827 (24)	0·6687 (3)	0·40503 (18)	0·0690 (20)
C(11)	1·09069 (22)	0·68749 (24)	0·33220 (17)	0·0611 (19)
C(12)	0·98492 (20)	0·66579 (21)	0·25369 (16)	0·0522 (17)
C(13)	0·97873 (20)	0·53800 (22)	0·22329 (16)	0·0516 (17)
C(13*A*)	1·04104 (25)	0·5098 (3)	0·16604 (20)	0·0791 (23)
N(14)	0·92345 (17)	0·46360 (17)	0·24950 (13)	0·0549 (14)
N(15)	0·91968 (17)	0·34518 (17)	0·22320 (14)	0·0563 (15)
C(16)	0·86267 (21)	0·26705 (22)	0·25296 (17)	0·0539 (17)
S(16)	0·84902 (7)	0·12425 (6)	0·22061 (5)	0·0684 (5)
N(17)	0·81777 (18)	0·31414 (19)	0·30642 (14)	0·0652 (16)
C(18)	0·75602 (22)	0·25938 (23)	0·35081 (16)	0·0564 (18)
C(19)	0·67541 (24)	0·3273 (3)	0·36499 (18)	0·0716 (21)
C(20)	0·6159 (3)	0·2816 (4)	0·41169 (21)	0·086 (3)
C(21)	0·6349 (3)	0·1684 (4)	0·44292 (20)	0·086 (3)
C(22)	0·7159 (3)	0·1008 (3)	0·43010 (19)	0·0805 (24)
C(23)	0·77782 (25)	0·14514 (25)	0·38406 (18)	0·0684 (20)

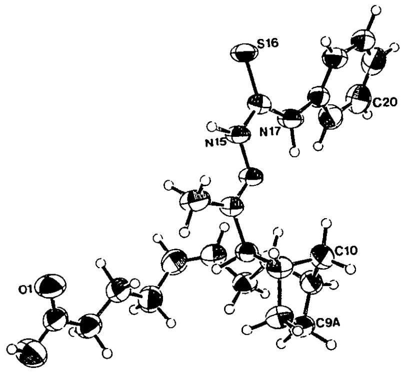


Fig. 1. EP 092. Non-H atoms are shown as 50% probability thermal ellipsoids.

Table 2. *Bond lengths* (Å), *angles* (°), *geometry* (Å *and* °) *around hydrogen bonds and selected torsion angles* (°), *with estimated standard deviations in parentheses*

C(1)—O(1)	1·197 (4)	C(11)—C(12)	1·558 (4)
C(1)—O(1*A*)	1·310 (4)	C(12)—C(13)	1·514 (4)
C(1)—C(2)	1·497 (5)	C(13)—C(13*A*)	1·495 (4)
C(2)—C(3)	1·500 (5)	C(13)—N(14)	1·276 (3)
C(3)—C(4)	1·527 (5)	N(14)—N(15)	1·395 (3)
C(4)—C(5)	1·488 (5)	N(15)—C(16)	1·351 (4)
C(5)—C(6)	1·313 (5)	C(16)—S(16)	1·681 (3)
C(6)—C(7)	1·499 (4)	C(16)—N(17)	1·342 (4)
C(7)—C(8)	1·537 (4)	N(17)—C(18)	1·414 (4)
C(8)—C(9)	1·538 (4)	C(18)—C(19)	1·380 (4)
C(8)—C(12)	1·550 (4)	C(18)—C(23)	1·387 (4)
C(9)—C(9*A*)	1·530 (4)	C(19)—C(20)	1·381 (5)
C(9)—C(10)	1·528 (4)	C(20)—C(21)	1·362 (5)
C(9*A*)—C(9*B*)	1·558 (5)	C(21)—C(22)	1·372 (5)
C(9*B*)—C(11)	1·538 (4)	C(22)—C(23)	1·390 (5)
C(10)—C(11)	1·528 (4)		

O(1)—C(1)—O(1*A*)	122·6 (3)	C(8)—C(12)—C(11)	102·55 (20)
O(1)—C(1)—C(2)	124·7 (3)	C(8)—C(12)—C(13)	116·88 (21)
O(1*A*)—C(1)—C(2)	112·7 (3)	C(11)—C(12)—C(13)	111·28 (21)
C(1)—C(2)—C(3)	114·4 (3)	C(12)—C(13)—C(13*A*)	116·31 (23)
C(2)—C(3)—C(4)	112·4 (3)	C(12)—C(13)—N(14)	118·05 (23)
C(3)—C(4)—C(5)	112·1 (3)	C(13*A*)—C(13)—N(14)	125·62 (25)
C(4)—C(5)—C(6)	128·2 (3)	C(13)—N(14)—N(15)	118·22 (21)
C(5)—C(6)—C(7)	127·6 (3)	N(14)—N(15)—C(16)	117·45 (21)
C(6)—C(7)—C(8)	111·18 (23)	N(15)—C(16)—S(16)	120·36 (20)
C(7)—C(8)—C(9)	116·10 (23)	N(15)—C(16)—N(17)	114·70 (24)
C(7)—C(8)—C(12)	113·20 (21)	S(16)—C(16)—N(17)	124·91 (21)
C(9)—C(8)—C(12)	103·10 (21)	C(16)—N(17)—C(18)	130·20 (23)
C(8)—C(9)—C(9*A*)	111·48 (24)	N(17)—C(18)—C(19)	117·14 (25)
C(8)—C(9)—C(10)	100·21 (23)	N(17)—C(18)—C(23)	122·85 (25)
C(9*A*)—C(9)—C(10)	101·67 (24)	C(19)—C(18)—C(23)	119·9 (3)
C(9)—C(9*A*)—C(9*B*)	103·08 (24)	C(18)—C(19)—C(20)	120·2 (3)
C(9*A*)—C(9*B*)—C(11)	102·69 (24)	C(19)—C(20)—C(21)	120·3 (3)
C(9)—C(10)—C(11)	94·44 (23)	C(20)—C(21)—C(22)	119·9 (3)
C(9*B*)—C(11)—C(10)	101·84 (23)	C(21)—C(22)—C(23)	120·9 (3)
C(9*B*)—C(11)—C(12)	107·14 (23)	C(18)—C(23)—C(22)	118·7 (3)
C(10)—C(11)—C(12)	101·92 (22)		

O(1*A*)—S(16)	3·147 (3)	C(1)—O(1)—H(151)	132·76 (24)
H(1*A*1)—S(16)	2·26 (3)	O(1)—H(151)—N(15)	169·90 (22)
O(1)—N(15)	2·989 (3)	O(1*A*)—H(1*A*1)—S(16)	168·6 (23)
O(1)—H(151)	1·919 (3)	H(1*A*1—S(16)—C(16)	109·0 (7)

C(9*B*)—C(11)—C(12)—C(13)	158·67 (23)	C(10)—C(11)—C(12)—C(13)	−94·80 (25)
C(8)—C(12)—C(13)—C(13*A*)	158·93 (24)	C(8)—C(12)—C(13)—N(14)	−22·4 (3)
C(11)—C(12)—C(13)—C(13*A*)	−83·8 (3)	C(6)—C(7)—C(8)—C(9)	−166·96 (24)
C(11)—C(12)—C(13)—N(14)	94·9 (3)	C(6)—C(7)—C(8)—C(12)	74·1 (3)
C(12)—C(13)—N(14)—N(15)	−178·53 (21)	C(7)—C(8)—C(9)—C(9*A*)	−57·8 (3)
C(13*A*)—C(13)—N(14)—N(15)	0·0 (4)	C(7)—C(8)—C(9)—C(10)	−164·72 (23)
C(13)—N(14)—N(15)—C(16)	178·65 (24)	N(14)—N(15)—C(16)—S(16)	177·01 (18)
N(14)—N(15)—C(16)—N(17)	−1·2 (3)	C(7)—C(8)—C(12)—C(11)	131·94 (23)
N(15)—C(16)—N(17)—C(18)	−178·21 (25)	C(7)—C(8)—C(12)—C(13)	−106·1 (3)
S(16)—C(16)—N(17)—C(18)	3·7 (4)	C(16)—N(17)—C(18)—C(23)	36·5 (4)

program *CALC* (Gould & Taylor, 1983) showed that all atoms of the thiocarbamoyl arm were planar with an r.m.s. deviation of 0·015 Å and the greatest deviation from the plane was 0·025 Å for the N(14) atom. Fig. 2 shows a stereodiagram of the molecular packing from which it can be seen that the benzene ring approaches C(9*A*) and C(9*B*) of the bicycloheptane moiety. The molecules arrange themselves as dimers across an inversion centre and are stabilized by hydrophobic interactions and by two hydrogen bonds. These occur by donation of the H atom on N(15) to the carboxyl oxygen, O(1), and of the H atom on the other carboxylic acid oxygen O(1*A*) to S(16). The associated bond distances and angles (Table 2) are in good agreement with tabulated values (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987).

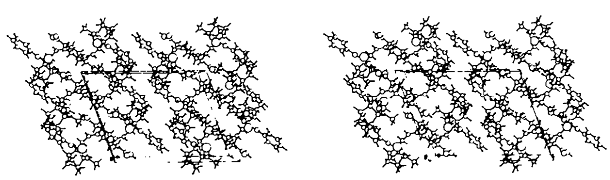


Fig. 2. Stereopacking of EP 092, *b*-axis projection.

It is not clear why the dramatic effect on the thromboxane receptor arises from this structure when it is compared with that of thromboxane B_2. The difference of the torsion angle between the bicycloheptane and the carboxyl arm confers no extra stability in the free molecule and may simply be the result of the hydrogen bonding. Clarification of the mode of action of these molecules must await the structure of the receptor.

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Acta Cryst. (1990). C**46**, 450–453

Structure of 9-Chloro-7-(2-chlorophenyl)-3,5-dihydro-[1,2,4]triazino[4,3-*a*][1,4]-benzodiazepin-2(1*H*)-one*

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Abstract. $C_{17}H_{12}Cl_2N_4O$, $M_r = 359{\cdot}2$, triclinic, $P\bar{1}$, $a = 8{\cdot}777\,(8)$, $b = 12{\cdot}715\,(4)$, $c = 14{\cdot}883\,(4)$ Å, $\alpha = 95{\cdot}69\,(2)$, $\beta = 83{\cdot}62\,(3)$, $\gamma = 93{\cdot}46\,(3)°$, $V = 1640{\cdot}5$ Å^3, $Z = 4$, $D_x = 1{\cdot}454$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 3{\cdot}99$ cm^{-1}, $F(000) = 736$, $T = 293$ K, $R = 0{\cdot}039$ for 2783 observed reflections. The seven-membered heterocyclic ring has a cycloheptatriene-like boat conformation with bow and stern angles 60·5 (8) and 33·8 (8)°, and 59·8 (8) and 35·9 (8)°, respectively, in the two independent molecules. The triazino ring is near planar in both molecules. The angles between the 7-phenyl ring and the fused benzo moiety are 88·9 (8) and 82·0 (8)°. Corresponding bond lengths and angles in the two molecules are generally similar and agree well with accepted values.

Introduction. The title compound (Szmuszkovicz, 1973) is related to the classical psychoactive 5-phenyl-1,4-benzodiazepin-2-ones such as diazepam, but differs from them in containing a six-membered hetero ring fused across the N(1)—C(2) bond of the parent system. Many of these diazepam derivatives exhibit a high degree of biological activity and the present compound binds to the benzodiazepine receptor *in vitro* about eight times more strongly than diazepam.

Experimental. Crystals were grown from amyl acetate. A crystal of size 0·3 × 0·4 × 0·7 mm was mounted on an Enraf–Nonius CAD-4 diffractometer and cell dimensions determined from the setting angles of 25 reflections in the range $10 < \theta < 16°$. Intensity data were measured with graphite-monochromated Mo $K\alpha$ radiation. 5121 unique reflections were scanned by ω–2θ scans up to $\theta = 24°$, 2783 $[I > 2{\cdot}5\sigma(I)]$ reflections were considered observed and used in the analysis, index range h −9 to 10, k −14 to 14, l 0 to 16. Two standard reflections measured every 2 h showed no significant variation in intensity. No absorption corrections were applied.

The structure was determined by direct methods and refined by least squares using anisotropic thermal parameters for the heavier atoms. H atoms were initially located from a difference synthesis and then

* Contribution from the Crystallography Unit, Universities of Aston and Birmingham.

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