

Acta Cryst. (1995). **C51**, 2673–2674

Tetrahydro-6-methyl-1*H*,3*H*-thieno[3,4-*b*]-pyrrol-2-one, an Analogue of Biotin

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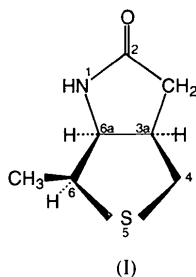
(Received 10 March 1995; accepted 30 June 1995)

Abstract

The title compound, $C_7H_{11}NOS$, an analogue of biotin, was prepared in order to further investigate the binding characteristics of biotin. The bicyclic ring system has pseudo-mirror symmetry with the tetrahydrothieno ring in an $S5\beta$ -envelope form with the methyl substituent in an equatorial position. The *cis*-fused pyrrolidone system is planar [non-H atoms coplanar within 0.03 (1) Å] and the dihedral angle between it and the four coplanar C atoms of the tetrahydrothieno ring is 119.0 (2)°. The C—S bond lengths are 1.808 (4) and 1.803 (4) Å, and the C—S—C angle is 88.8 (2)°.

Comment

The structure determination of tetrahydro-6-methyl-1*H*,3*H*-thieno[3,4-*b*]pyrrol-2-one, (1), a crippled analogue of biotin, (2), which is a cofactor for the enzyme acetyl coA carboxylase, was carried out in order to further elucidate the binding characteristics of the acetyl coA carboxylase active site (Green, 1963; Knowles, 1989).



A perspective view of the 3*aS*,6*aS*,6*S* enantiomer is illustrated in Fig. 1. The bicyclic tetrahydrothienopyrrolidone system has a conformation similar to that reported for the tetrahydrothienoimidazolone system in biotin (DeTitta, Edmonds, Stallings & Donohue,

1976). The bicyclic moiety has pseudo-mirror symmetry about a plane containing atoms C2 and S5, and normal to the C3*a*—C6*a* bond, with an asymmetry parameter (Duax & Norton, 1975) C_s^2 of 0.7° (*cf.* 3.0° for biotin). The four C atoms of the tetrahydrothieno ring are coplanar within experimental limits and atom S5 lies 0.91 (1) Å from this plane (*cf.* 0.87 Å in biotin). The pseudo-rotation parameters (Altona, Geise & Romers, 1968) for the ring are $\Delta = -35.2^\circ$ and $\psi = 54.0^\circ$. The bond lengths and angles given in Table 2 are typical for such systems and similar to the analogous values reported for biotin (DeTitta *et al.*, 1976). As observed in the structure of a polymorph of diacetamide (Kuroda, Taira, Uno & Osaki, 1975), intermolecular hydrogen bonds in which the N atom donates its proton to the C2 carbonyl O atom link the molecules into centrosymmetric cyclic dimers, graph set $R_2^2(8)$ (Etta, 1990); the N1—H1...O2(2-x, 1-y, 2-z) and H1...O2 distances are 0.88 (4), 2.884 (3) and 2.00 (4) Å, respectively, with an N1—H1...O2 angle of 173 (3)°.

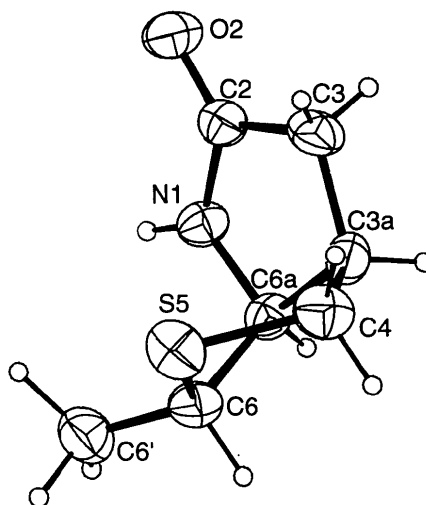


Fig. 1. The molecular structure of the 3*aS*,6*aS*,6*S* enantiomer with displacement ellipsoids scaled to 50% probability. The H atoms are denoted by spheres of arbitrary radii.

Experimental

Crystals of tetrahydro-6-methyl-1*H*,3*H*-thieno[3,4-*b*]pyrrol-2-one suitable for an X-ray study were prepared according to the method reported by Henderson, O'Connor, Rendina, Savage & Simpson (1995). The compound was recrystallized from a 1:1 chloroform–ethyl acetate solution.

Crystal data

$C_7H_{11}NOS$
 $M_r = 157.2$

Cu $K\alpha$ radiation
 $\lambda = 1.5418 \text{ \AA}$

Triclinic	Cell parameters from 25 reflections
$P\bar{1}$	$\theta = 4.1\text{--}65.0^\circ$
$a = 11.119(1)\text{ \AA}$	$\mu = 3.22\text{ mm}^{-1}$
$b = 6.368(1)\text{ \AA}$	$T = 292(1)\text{ K}$
$c = 5.582(1)\text{ \AA}$	Prismatic
$\alpha = 101.60(1)^\circ$	$0.410 \times 0.205 \times 0.130\text{ mm}$
$\beta = 90.43(1)^\circ$	Pale yellow
$\gamma = 102.40(1)^\circ$	
$V = 377.6(1)\text{ \AA}^3$	
$Z = 2$	
$D_x = 1.383\text{ Mg m}^{-3}$	

Data collection

Rigaku AFC diffractometer	$R_{\text{int}} = 0.029$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 65.04^\circ$
Absorption correction:	$h = -12 \rightarrow 12$
<i>SHELX76</i> Gaussian (Sheldrick, 1976)	$k = -7 \rightarrow 7$
$T_{\text{min}} = 0.460$, $T_{\text{max}} = 0.685$	$l = 0 \rightarrow 6$
1182 measured reflections	3 standard reflections monitored every 100 reflections
844 independent reflections	intensity decay: $>1.5\%$
797 observed reflections [$F > 3\sigma(F)$]	

Refinement

Refinement on F^2	$\Delta\rho_{\text{max}} = 0.254\text{ e \AA}^{-3}$
$R = 0.047$	$\Delta\rho_{\text{min}} = -0.245\text{ e \AA}^{-3}$
$wR = 0.066$	Extinction correction:
$S = 3.01$	$F_c = F(1 - \chi/\sin\theta)$
847 reflections	Extinction coefficient:
136 parameters	$\chi = 0.049(16) \times 10^{-6}$
All H-atom parameters refined	Atomic scattering factors from <i>International Tables for Crystallography</i> (1992), Vol. C, Tables 4.2.6.8 and 6.1.1.4)
$w = 1/[\sigma^2(F) + 0.00036F^2]$	
$(\Delta/\sigma)_{\text{max}} = 0.003$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
N1	0.8949 (2)	0.4911 (4)	0.7407 (5)	0.032 (1)
C2	0.9108 (3)	0.2847 (5)	0.7003 (6)	0.033 (1)
O2	0.9751 (2)	0.2123 (4)	0.8316 (5)	0.044 (1)
C3	0.8397 (4)	0.1571 (6)	0.4696 (8)	0.043 (1)
C3a	0.7706 (3)	0.3128 (6)	0.3763 (7)	0.036 (1)
C4	0.6305 (4)	0.2380 (6)	0.3769 (8)	0.047 (1)
S5	0.5905 (1)	0.3370 (1)	0.6862 (2)	0.0456 (5)
C6	0.6946 (3)	0.5965 (5)	0.6854 (7)	0.037 (1)
C6'	0.7174 (4)	0.7511 (7)	0.9336 (8)	0.050 (1)
C6a	0.8104 (3)	0.5350 (6)	0.5665 (7)	0.032 (1)

Table 2. Selected geometric parameters (\AA , $^\circ$)

N1—C2	1.338 (4)	C3a—C6a	1.564 (5)
N1—C6a	1.454 (4)	C4—S5	1.808 (4)
C2—O2	1.237 (5)	S5—C6	1.803 (4)
C2—C3	1.494 (5)	C6—C6'	1.514 (5)
C3—C3a	1.541 (6)	C6—C6a	1.542 (5)
C3a—C4	1.528 (5)		
C2—N1—C6a	115.4 (2)	C3a—C4—S5	105.7 (3)
O2—C2—N1	125.6 (2)	C6—S5—C4	88.8 (2)
O2—C2—C3	125.6 (3)	C6'—C6—C6a	115.4 (3)
N1—C2—C3	109.0 (2)	C6'—C6—S5	114.1 (3)
C2—C3—C3a	106.9 (3)	C6a—C6—S5	104.9 (2)

C4—C3a—C3	113.0 (3)	N1—C6a—C6	112.7 (2)
C4—C3a—C6a	107.7 (3)	N1—C6a—C3a	104.4 (2)
C3—C3a—C6a	104.3 (3)	C6—C6a—C3a	108.8 (3)

Data collection: Rigaku AFC diffractometer software. Cell refinement: Rigaku AFC diffractometer software. Data reduction: Rigaku AFC diffractometer software. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*. Computer: VAX 8800.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1030). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). **C51**, 2674–2676

1,1-Diphenyl-3-propyn-1-ol

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(Received 7 April 1995; accepted 20 June 1995)

Abstract

The aromatic ring planes in the title compound, C₁₅H₁₂O, are almost mutually orthogonal, forming a dihedral angle of magnitude 94.15 (5)°. The C≡C triple