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*Acta Cryst.* (1992). C**48**, 1326–1327

# Structure of DL-Desthiobiotin

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(*Received* 31 *May* 1991; *accepted* 19 *November* 1991)

**Abstract.** $C_{10}H_{18}N_2O_3$, $M_r = 214.26$, monoclinic, $P2_1/a$, $a = 9.149$ (2), $b = 10.934$ (1), $c = 12.064$ (1) Å, $\beta = 93.14$ (1)°, $V = 1205.1$ (5) Å$^3$, $Z = 4$, $D_m = 1.178$ (1), $D_x = 1.181$ Mg m$^{-3}$, $\lambda$(Cu $K\alpha$) = 1.54178 Å, $\mu = 0.685$ mm$^{-1}$, $F(000) = 464$, $T = 296$ K, final $R = 0.048$ for 1521 reflections [$I > 3\sigma(I)$]. The molecule has a fully extended caproic acid side chain with a *trans* configuration. The imidazole ring is protonated; three kinds of hydrogen bonds are formed among the imino groups and the carboxyl group.

**Experimental.** Crystals of desthiobiotin were obtained from water as needles, 0.2 × 0.2 × 0.5 mm; Rigaku AFC5R automated four-circle diffractometer with graphite-monochromated Cu $K\alpha$ radiation; lattice parameters determined from $2\theta$ values of 25 reflections ($78.4 < 2\theta < 79.9°$); intensity data to $2\theta = 120.1°$, $\omega$–$2\theta$ scan, scan speed 32.0° ($\omega$) min$^{-1}$, scan width $(1.78 + 0.30 \tan\theta)°$; ratio of peak counting time to background counting time 2:1; $h$ 0→10, $k$ 0→12, $l$ −13→13, 2038 reflections measured of which 1521 with $I > 3\sigma(I)$ were used for the analysis; three reference reflections monitored at an interval of 100 reflections showed no crystal deterioration; Lorentz, polarization and absorption corrections (max. and min. transmission factors 0.96, 1.00), structure solved by direct methods with *MITHRIL* (Gilmore, 1984) and *DIRDIF* (Beurskens, 1984), refined by least squares with anisotropic thermal parameters for all non-H atoms; H atoms located from difference Fourier map, included in refinement with isotropic thermal parameters; $\sum w(|F_o| - |F_c|)^2$ minimized, $w = 4F_o^2/\sigma^2(F_o^2)$, number of parameters: 208; final $R = 0.048$, $wR = 0.067$; $(\Delta/\sigma)_{max} = 0.03$, $S = 2.80$, maximum and minimum peaks in the final difference Fourier map 0.18 and −0.15 e Å$^{-3}$; atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV); all numerical calculations performed using the *TEXSAN* crystallographic software package of Molecular Structure Corporation (1985). Final atomic parameters of non-H atoms are listed in Table 1.* Selected bond lengths, angles and hydrogen bonds are listed in Table 2. A perspective view of desthiobiotin is shown in Fig. 1 with the atomic numbering scheme.

**Related literature.** The title compound is a precursor of biotin (Vitamin H) which is a growth factor for both yeast and humans and functions as a coenzyme for carboxylation reaction (Tanaka, Izumi & Yamada, 1988). Desthiobiotin also acts like biotin for microorganisms such as *Bacillus subtilis* (Izumi, Kano, Inagaki, Kawase, Tani & Yamada, 1981). The

* Lists of structure factors, anisotropic thermal parameters for non-H atoms, and coordinates and isotropic thermal parameters for H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54877 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0509]

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Table 1. *Atomic coordinates for non-H atoms with e.s.d.'s in parentheses*

$B_{eq} = \frac{4}{3}(B_{11}a^2 + B_{22}b^2 + B_{33}c^2 + B_{13}ac\cos\beta)$.

| | *x* | *y* | *z* | $B_{eq}$ (Å$^2$) |
|---|---|---|---|---|
| O(2′) | 0.4733 (2) | 0.4823 (1) | 0.3456 (1) | 4.77 (7) |
| O(10*A*) | 0.8293 (3) | −0.0014 (2) | 1.1491 (1) | 6.9 (1) |
| O(10*B*) | 0.9365 (3) | 0.1647 (3) | 1.0928 (2) | 10.9 (2) |
| N(1′) | 0.6120 (2) | 0.3172 (2) | 0.2987 (2) | 5.0 (1) |
| N(3′) | 0.5941 (2) | 0.3583 (2) | 0.4734 (2) | 4.6 (1) |
| C(2) | 0.6820 (3) | 0.1701 (2) | 0.5674 (2) | 4.7 (1) |
| C(2′) | 0.5526 (2) | 0.3934 (2) | 0.3708 (2) | 3.9 (1) |
| C(3) | 0.7004 (3) | 0.2596 (2) | 0.4738 (2) | 4.3 (1) |
| C(4) | 0.6764 (3) | 0.2106 (2) | 0.3539 (2) | 4.5 (1) |
| C(5) | 0.5791 (4) | 0.0993 (3) | 0.3387 (2) | 5.9 (1) |
| C(6) | 0.7353 (4) | 0.2188 (3) | 0.6808 (2) | 5.6 (1) |
| C(7) | 0.7048 (4) | 0.1319 (3) | 0.7741 (2) | 5.5 (1) |
| C(8) | 0.7865 (4) | 0.1629 (3) | 0.8830 (2) | 6.3 (2) |
| C(9) | 0.7573 (5) | 0.0700 (3) | 0.9715 (2) | 7.3 (2) |
| C(10) | 0.8507 (4) | 0.0844 (3) | 1.0758 (2) | 6.1 (1) |

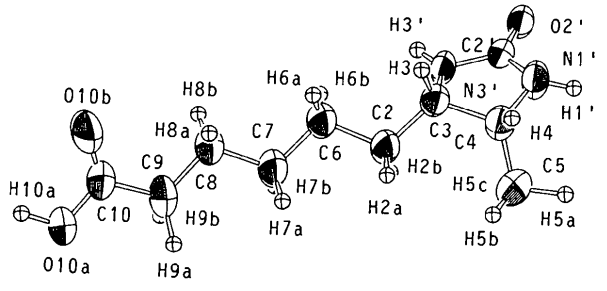


Fig. 1. Perspective view of desthiobiotin with the atomic numbering used.

crystal structure of (±)-desthiobiotin has previously been determined (Chen, Parthasarathy & DeTitta, 1976). Their crystal has a different packing system from that of the present one, but the molecular conformations in both crystals are similar. The crystal structure of biotin has also been reported (Traub, 1956; Bonnemere, Hamilton, Steinrauf & Knappe, 1965).

Table 2. *Bond lengths* (Å) *and angles* (°) *between non-H atoms, and hydrogen bonds* (Å)

| | | | |
|---|---|---|---|
| O(2′)—C(2′) | 1.241 (3) | C(2)—C(6) | 1.523 (3) |
| O(10*A*)—C(10) | 1.311 (3) | C(3)—C(4) | 1.548 (3) |
| O(10*B*)—C(10) | 1.188 (3) | C(4)—C(5) | 1.513 (4) |
| N(1′)—C(2′) | 1.341 (3) | C(6)—C(7) | 1.511 (4) |
| N(1′)—C(4) | 1.452 (3) | C(7)—C(8) | 1.514 (4) |
| N(3′)—C(2′) | 1.332 (3) | C(8)—C(9) | 1.508 (4) |
| N(3′)—C(3) | 1.453 (3) | C(9)—C(10) | 1.490 (4) |
| C(2)—C(3) | 1.510 (3) | | |

| | | | |
|---|---|---|---|
| C(2′)—N(1′)—C(4) | 111.7 (2) | N(1′)—C(4)—C(5) | 111.6 (2) |
| C(2′)—N(3′)—C(3) | 111.9 (2) | C(3)—C(4)—C(5) | 116.3 (2) |
| C(3)—C(2)—C(6) | 113.7 (2) | C(2)—C(6)—C(7) | 112.7 (2) |
| O(2′)—C(2′)—N(1′) | 125.4 (2) | C(6)—C(7)—C(8) | 113.8 (3) |
| O(2′)—C(2′)—N(3′) | 125.9 (2) | C(7)—C(8)—C(9) | 111.5 (3) |
| N(1′)—C(2′)—N(3′) | 108.6 (2) | C(8)—C(9)—C(10) | 114.3 (3) |
| N(3′)—C(3)—C(2) | 112.4 (2) | O(10*A*)—C(10)—O(10*B*) | 122.3 (2) |
| N(3′)—C(3)—C(4) | 101.2 (2) | O(10*A*)—C(10)—C(9) | 113.2 (3) |
| C(2)—C(3)—C(4) | 117.3 (2) | O(10*B*)—C(10)—C(9) | 124.5 (3) |
| N(1′)—C(4)—C(3) | 100.6 (2) | | |

| *D* (at *x*,*y*,*z*) | *A* | *D*···*A* |
|---|---|---|
| O(10*A*) | O(2′)$^{i}$ | 2.657 (2) |
| N(1′) | O(10*B*)$^{ii}$ | 2.889 (3) |
| N(3′) | O(2′)$^{iii}$ | 2.887 (2) |

Symmetry code: (i) $\frac{3}{2}+x, \frac{1}{2}-y, 1+z$; (ii) $\frac{1}{2}+x, \frac{1}{2}-y, -1+z$; (iii) $-1-x, -1-y, -1-z$.

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*Acta Cryst.* (1992). C**48**, 1327–1329

# Monoprotonated Perchlorate Salt of Tris(2-pyridyl)methanol


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(*Received* 12 *July* 1991; *accepted* 21 *November* 1991)


**Abstract.** 2-[Bis(2-pyridyl)hydroxymethyl]pyridinium perchlorate, $C_{16}H_{14}N_3O^+.ClO_4^-$, $M_r = 363.8$, monoclinic, $P2_1/c$, $a = 12.301$ (3), $b = 13.300$ (4), $c = 10.817$ (3) Å, $\beta = 110.94$ (2)°, $V = 1652.8$ (8) Å$^3$, $Z = 4$, $D_x = 1.462$ Mg m$^{-3}$, Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å, $\mu = 0.26$ mm$^{-1}$, $F(000) = 752$, $T = 293$ (1) K, $R = 0.053$ for 2504 observed reflections. The C—O bond length is 1.407 (4) Å; the C—C(OH)


0108-2701/92/071327-03$06.00