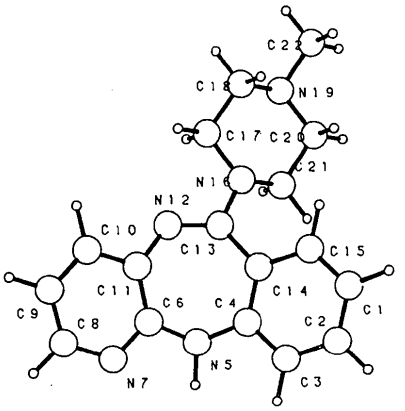

Fig. 1. Vue en perspective de la molécule *A* et numérotation des atoms.

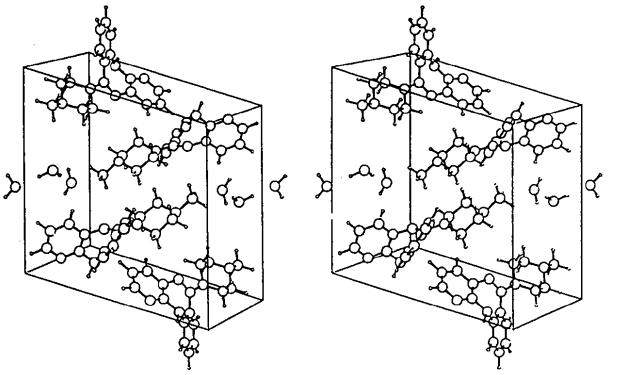

Fig. 2. Vue stéréoscopique de la structure suivant 0*x*.

finale: $-0{,}32$ et $0{,}39$ e Å^{-3}. Les paramètres atomiques sont donnés dans le Tableau 1.* La liste des distances et des angles des liaisons se trouve dans le Tableau 2. La Fig. 1 montre une vue en perspective de la molécule *A* du composé avec la numérotation atomique. Celle-ci est incrémentée de 50 pour les atomes de la molécule *B* dans le Tableau 1. La Fig. 2 représente la structure cristalline.

Littérature associée. La structure du 6-(4-méthylpipérazin-1-yl)-11*H*-pyrido[2,3-*b*][1,4]benzodiazépine 1,5-hydrate a été réalisée dans le cadre de notre étude sur les récepteurs à dopamine. Voir par exemple les structures du 11-formyl-5-(4-méthylpipérazin-1-yl)-11*H*-pyrido[2,3-*b*][1,5]benzodiazépine et du 6-(4-méthylpipérazin-1-yl)-11-méthyl-11*H*-pyrido[2,3-*b*]-[1,4]benzodiazépine (Dupont, Englebert, Dideberg, Liégeois & Delarge, 1991). Des analogues hétérocycliques originaux sont en voie d'expérimentation.

Les auteurs remercient M. M. Vermeire pour l'assistance technique et le FNRS pour le crédit alloué au programme de recherche dont ce travail constitue une partie.

* Les listes des facteurs de structure calculeés et observés, les facteurs d'agitation thermique anisotrope, les coordonnées des atomes H et des paramètres des plans moyens ont été déposées au dépôt d'archives de la British Library Document Supply Centre (Supplementary Publication No. SUP 54869: 14 pp.). On peut en obtenir des copies en s'adressant à: The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre. [Référence de CIF: PA0256]

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Structure of 2-Hydroxy-1-naphthaldehyde

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Abstract. $C_{11}H_8O_2$, $M_r = 172.18$, monoclinic, $P2_1/n$, $a = 5.630$ (1), $b = 9.341$ (2), $c = 15.531$ (3) Å, $\beta = 98.40$ (1)°, $V = 808.0$ (3) Å^3, $Z = 4$, $D_m = 1.41$ (flotation in CH_3I/benzene), $D_x = 1.415$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 0.75$ mm^{-1}, $F(000) = 360$, room temperature, final $R = 0.051$ for 1074 observed reflections (of 1492 unique data). An intramolecular hydrogen bond is observed for the title

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compound with O1···O2 = 2.567 (3), H1···O2 = 1.59 (1), O1—H1 = 1.070 (6) Å and O1—H1···O2 = 148.9 (5)°. The six-membered rings *A* and *B* of the naphthalene molecule are planar (within the 2.5σ range) with an angle between normals to the *A* and *B* planes of 0.38 (9)°. The O1, O2, C11 and H1 atoms deviate from the best plane of the *A* ring by −0.036 (2), 0.029 (2), 0.035 (2) and 0.149 (5) Å, respectively. The H atom in the hydrogen bridge has a very large *U* value [0.109 (1) Å^2]. This suggests proton motion in the asymmetric O—H—O hydrogen bridge.

Experimental. The title compound was obtained from Aldrich Chemical Co. Crystal dimensions 0.1 × 0.15 × 0.35 mm. All data were obtained from a KM-4 automated four-circle diffractometer. Final lattice parameters were from least-squares refinement of 25 reflections ($10 < \theta < 40°$); no absorption correction was applied; $\theta < 75°$ (*h*: 0/7, *k*: 0/12, *l*: −19/19); ω–1.7θ scan technique; Cu $K\alpha$ radiation at room temperature; intensities of three standard reflections were monitored every 50 reflections and showed no significant fluctuations; 1492 unique reflections were measured, 1074 were observed with $I > 3\sigma(I)$; $R_{int} = 0.068$. The structure was solved by direct methods (*SHELXS*86; Sheldrick, 1986) with $R(E) = 0.32$. An *E* map provided positions for all non-H atoms. Positions of all H atoms were taken from a $\Delta\rho$ map and refined. Refinement (on F^2) by full-matrix least squares with anisotropic temperature factors for all non-H atoms converged to $R = 0.051$ {with $w = 1/[\sigma(F)^2 + 0.00055F^2]$, $wR = 0.062$} with the empirical extinction correction coefficient $g = 0.004$; 151 parameters were varied; for all parameters $\Delta/\sigma < 0.003$. The minimum and maximum peaks in the final $\Delta\rho$ map were −0.18 and 0.20 e Å^{-3}; atomic scattering factors were taken from *SHELXTL-PC* (Sheldrick, 1989). All calculations were performed using the *SHELXTL-PC* system and the *CSU* program (Vicković, 1988) on the PC computer. A view of the molecule with the atomic numbering is presented in Fig. 1, coordinates

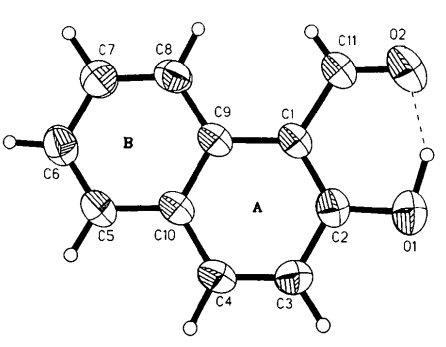

Fig. 1. View of the molecule showing atomic numbering scheme.

Table 1. *Final positional atomic parameters of the non-*H *atoms* (× 10^4) *and equivalent isotropic temperature factors* (Å^2 × 10^4) *with e.s.d.'s in parentheses*

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

	x	*y*	*z*	U_{eq}
O1	2259 (3)	924 (2)	534.7 (9)	617 (3)
O2	−1255 (3)	2018 (2)	1192.1 (10)	733 (3)
C1	−335 (3)	2720 (2)	−189.0 (10)	404 (3)
C2	1565 (3)	1775 (2)	−156.8 (11)	433 (3)
C3	2913 (3)	1668 (2)	−850.2 (12)	487 (3)
C4	2349 (3)	2517 (2)	−1563.6 (11)	444 (3)
C5	−136 (3)	4382 (2)	−2377.0 (11)	463 (3)
C6	−1967 (3)	5337 (2)	−2444.6 (12)	515 (3)
C7	−3327 (4)	5467 (2)	−1761.4 (13)	537 (3)
C8	−2813 (3)	4642 (2)	−1031.3 (11)	490 (3)
C9	−939 (3)	3631 (2)	−937.7 (10)	375 (3)
C10	442 (3)	3514 (2)	−1631.9 (10)	380 (3)
C11	−1735 (3)	2757 (2)	557.7 (11)	491 (3)

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

O1—C2	1.348 (2)	C2—C1—C9	119.8 (2)
O2—C11	1.201 (2)	C2—C1—C11	118.7 (2)
C1—C2	1.382 (3)	C9—C1—C11	121.6 (2)
C1—C9	1.441 (2)	O1—C2—C1	122.6 (2)
C1—C11	1.495 (2)	O1—C2—C3	116.2 (2)
C2—C3	1.408 (2)	C1—C2—C3	121.2 (2)
C3—C4	1.363 (2)	C2—C3—C4	119.6 (2)
C4—C10	1.414 (3)	C3—C4—C10	121.9 (2)
C5—C6	1.356 (3)	C6—C5—C10	121.6 (2)
C5—C10	1.411 (2)	C5—C6—C7	119.4 (2)
C6—C7	1.403 (3)	C6—C7—C8	120.5 (2)
C7—C8	1.366 (2)	C7—C8—C9	121.9 (2)
C8—C9	1.408 (3)	C1—C9—C8	124.3 (2)
C9—C10	1.422 (2)	C1—C9—C10	118.4 (2)
		C8—C9—C10	117.3 (2)
		C4—C10—C5	121.4 (2)
		C4—C10—C9	119.2 (2)
		C5—C10—C9	119.4 (2)
		O2—C11—C1	122.8 (2)

are given in Table 1, bond distances and angles in Table 2.*

Related literature. This study forms part of our work on copper(II) complexes with Schiff bases obtained from 2-hydroxy-1-naphthaldehyde and aliphatic aminoalcohols (Maniukiewicz & Bukowska-Strzyżewska, 1990; Bukowska-Strzyżewska & Maniukiewicz, 1992). In the naphthalene ring the bond lengths and angles are similar to those in the above papers. In contrast to the naphthalene molecule itself (Brock & Dunitz, 1982), the 2-hydroxy-1-naphthaldehyde and its derivatives show elongation of C1—C9 and C1—C2 bonds from 1.374 (2) to 1.382 (3)–1.415 (3) Å for C1—C9 and from 1.423 (2) to 1.441 (2)–1.452 (4) Å for C1—C2.

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54859 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL0517]

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Structure of DL-Desthiobiotin

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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}, λ(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 2θ values of 25 reflections ($78.4 < 2\theta < 79.9°$); intensity data to $2\theta = 120.1°$, ω–2θ scan, scan speed 32.0° (ω) 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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