Table I. Crystal Data for $C_7H_{11}NOS$, Oxazolethione 3

mol form	$C_7H_{11}NOS$
M_r	157.23
a	19.142 (7) Å
b	6.197 (3) Å
c	7.400 (3) Å
β	100.42 (3)°
V	863.3 (6) Å^3
$F(000)$	336
μ(Mo K$_\alpha$)	2.978 cm^{-1}
λ(Mo K$_\alpha$)	0.71069 Å
D_{calcd}	1.210 g cm^{-3}
Z	4
obsd refln	829
R	5.1%
space group	$P2_1/n$

155.4 ppm in 4, and its spin–lattice relaxation time was so long that a 10-s delay between acquisitions was required to detect the C(2) signal.

Formation of the oxazolethione was quite unexpected, for the procedure[1] is a standard method for 2-mercaptoimidazoles. In a separate experiment the residue from reaction of 1-bromopinacolone with ammonia was treated with cold aqueous KOH and extracted with ether. Recrystallization of the recovered solid from petroleum ether gave a crystalline product, mp 90–91 °C. ^{1}H and ^{13}C NMR spectra were consistent with 1-aminopinacolone as the major component in a mixture of compounds. Reaction of the crystalline mixture with KSCN and HCl as before also gave oxazolethione 3.

Experimental Section

IR spectra (KBr) were recorded with a Perkin-Elmer 681 spectrophotometer. ^{1}H NMR spectra at 300 MHz and ^{13}C NMR spectra at 75.43 MHz were recorded in $CDCl_3$ with a Varian XL-300 spectrometer. ^{13}C NMR spectra at 25.2 MHz were obtained with a Varian XL-100(15) instrument equipped with a Nicolet TT-100 PFT accessory. Mass spectral analyses were done with a CEC Model 21-110B high resolution double focusing mass spectrometer with a Data General DS-50S data system at 70 eV. Elemental analyses were carried out by Galbraith Laboratories (Knoxville, TN).

4-*tert*-Butyl-2(3*H*)-oxazolethione (3). To 500 mL of liquid ammonia in a two-neck 1-L round-bottom flask equipped with a cold finger at −78 °C was added 45 mL (0.33 mol) of 1-bromopinacolone (Fluka, 99% pure by GC). The reaction mixture was allowed to warm to −20 °C and stirred for 4 h. The excess ammonia evaporated slowly. Final traces of ammonia were removed by using a vacuum pump. The residual pale yellow solid was dissolved in 190 mL of ethanol, 210 mL (0.42 mol) of 2 M HCl was added, and 39.46 g (0.406 mol) of KSCN (Aldrich, 99+% pure) was added. The mixture was refluxed at 80 °C for 20 h under argon, cooled to room temperature, and added to ice water while being stirred well. The precipitate was filtered, washed with water, and air-dried to yield 33.3 g of solid. Flash chromatography of the solid over silica gel (Baker, 40-μm diameter) using a gradient mixture of ethyl acetate–petroleum ether as eluent gave 19.2 g (37% yield) of 4-*tert*-butyl-2(3*H*)-oxazolethione (3): mp 145–147 °C; IR 3300–2700 br, 3180, 3060, 2960, 1640, 1495, 1470, 1460, 1255, 1175, 1130, 1060, 980, 940, 935, 735, and 640 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 12.59 (1 H, br), 7.04 (1 H, s), and 1.30 (9 H, s); ^{13}C NMR ($CDCl_3$, 75.43 MHz) δ 178.60 s, 140.86 s, 130.78 d, 29.94 s, and 28.65 q; MS, m/z 157.0609 (M$^+$, 100%); calcd for $C_7H_{11}NOS$ 157.0561. Anal. Calcd for $C_7H_{11}NOS$: C, 53.47; H, 7.05; N, 8.91; O, 10.18; S, 20.39. Found: C, 53.40; H, 6.85; N, 8.89; O, 10.39; S, 20.67.

2,2′-Bis(4-*tert*-butyloxazolyl) Disulfide (4). To 16.93 g (0.108 mol) of 4-*tert*-butyl-2(3*H*)-oxazolethione (3) in 400 mL of toluene was added 18.89 g (0.217 mol) of active MnO_2 (Aldrich). The mixture was stirred at room temperature under argon for 25 h and filtered through a bed of silica gel. The filtrate was evaporated to give 14.1 g of a gummy residue. Flash chromatography over silica gel (Baker, ~40-μm diameter) using 2% ethyl acetate in petroleum ether as eluent gave 13.1 g (78% yield) of 2,2′-bis(4-*tert*-butyloxazolyl) disulfide (4): mp 68–70 °C; IR 3150, 3110, 2970, 2930, 2910, 2870, 1610 br w, 1570, 1465, 1450, 1365, 1360, 1210, 1175, 1140, 1080, 970, 945, 935, 805, 670, 665, and 605 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 7.47 (2 H, s) and 1.26 (18 H, s); ^{13}C NMR ($CDCl_3$, 25.2 MHz) δ 155.39 s, 153.07 s, 135.84 d, 31.18 s, and 29.03 q. MS, m/z 312.1011 (M$^+$, 24%); calcd for $C_{14}H_{20}N_2O_2S_2$ 312.0966. Anal. Calcd for $C_{14}H_{20}N_2O_2S_2$: C, 53.82; H, 6.45; N, 8.97; O, 10.24; S, 20.52. Found: C, 54.02; H, 6.37; N, 8.97; O, 10.00; S, 20.80.

X-ray Analysis of 4-*tert*-Butyl-2(3*H*)-oxazolethione (3). A crystal of 3 was mounted on a Syntex P3 automated diffractometer. Unit cell dimensions (Table I) were determined by least-squares refinement of the best angular positions for 15 independent reflections ($2\theta > 15°$) during normal alignment procedures using molybdenum radiation ($\lambda = 0.71069$ Å). Data (2348 points) were collected at room temperature by using a variable scan rate, a θ–2θ scan mode, and a scan width of 1.2° below Kα_1 and 1.2° above Kα_2 to a maximum 2θ value of 60.0°. Backgrounds were measured at each side of the scan for a combined time equal to the total scan time. The intensities of three standard reflections were remeasured after every 97 reflections, and as the intensities of these reflections showed less than 6% variation, corrections for decomposition were deemed unnecessary. Data were corrected for Lorentz, polarization, and background effects. After removal of redundant and space group forbidden data, 829 points were considered observed [$I > 3.0\ \sigma(I)$]. The structure was solved by using MULTAN80[4] to locate heavy atom positions. Successive cycles of least-squares refinement followed by difference Fourier synthesis allowed location of the remainder of the non-hydrogen atoms. Refinement of scale factor, positional, and anisotropic thermal parameters for all non-hydrogen atoms was carried out to convergence. Hydrogen positions were apparent from a final difference Fourier and were refined in the final cycles of least squares along with their isotropic thermal parameters.[5] The final cycle of refinement – [functional minimized $\sum(|F_o| - |F_c|)^2$] led to a final agreement factor, $R = 5.1\%$, $R = \sum(||F_o| - |F_c||/\sum|F_o|) \times 100$. Anomalous dispersion corrections were made for S. The scattering factors were taken from Cromer and Mann.[6] Unit weights were used until the final cycles of refinement, when a weight = $1/\sigma F$ was introduced. $R_w = 6.9$.

Acknowledgment. We thank the U.S. Army Research Office of the support of contract DAAG-82-K-0133. The 300-MHz NMR spectrometer was supported in part by National Science Foundation Grant CHE-8106157.

Supplementary Material Available: Tables II–V listing positional parameters, thermal parameters, distances from the plane, and bond angles and distances for compound 3 (4 pages). Ordering information is given on any current masthead page. A listing of calculated and observed structure factors is availabile from W.T.F.

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3,4-Dihydrobenz[*f*]isoquinoline and 3,4-Dihydrobenz[*g*]isoquinoline

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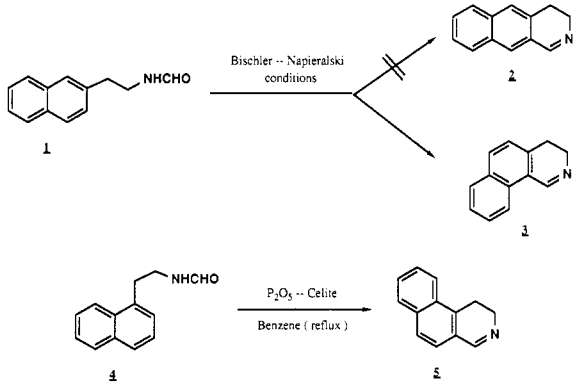
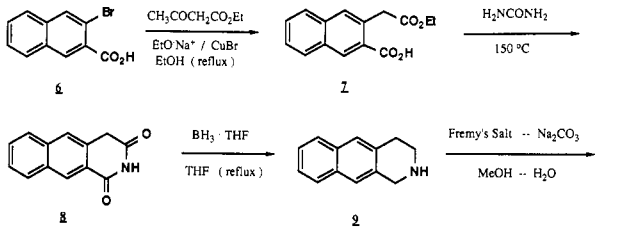
Previous attempts to prepare the linear isoquinoline 2 by Bischler–Napieralski cyclization of 2-(2-formamidoylethyl)naphthalene (1) resulted only in the formation of 3,4-dihydrobenz[*h*]isoquinoline (3).[1] When blocking

groups were introduced at C-1 the cyclocondensation failed or produced **3** by ipso displacement. The preparation of 3,4-dihydrobenz[*f*]isoquinoline (**5**) by Bischler–Napieralski cyclization of formamide **4** has been reported to fail under a variety of conditions.[2] An alternative multistep procedure was developed starting with 1-(2-hydroxyethyl)-naphthalene.[3] In our hands this procedure was difficult and required several tedious separations.



We have developed a four-step synthesis of 3,4-dihydrobenz[*g*]isoquinoline (**2**) from 2-bromo-3-carboxynaphthalene (**6**) and have succeeded in preparing 3,4-dihydrobenz[*f*]isoquinoline (**5**) from formamide **4** using improved Bischler–Napieralski conditions.[4,8]



Copper-catalyzed displacement of bromide from 2-bromo-3-carboxynaphthalene (**6**)[5] with the sodium salt of ethyl acetoacetate and concomitant loss of acetate gives homophthalate half-ester **7**.[6] Reaction of half-ester **7** with molten urea at 150 °C for 45 min provides the glutarimide **8** in good yield.[7] Reduction of **8** with borane in THF gives the tetrahydropyridine **9** and subsequent oxidation of **9** with Fremy's salt[9] provides 3,4-dihydrobenz[*g*]isoquinoline (**2**) in 70% to 80% yield.

The reported success of phosphorus pentoxide–Celite as a reagent for the Bischler–Napieralski reaction[8] encouraged us to apply this methodology to the cyclization of **4**. Accordingly when a solution of formamide **4** in chloroform is added to a mechanically stirred, refluxing mixture of phosphorus pentoxide–Celite in benzene, slowly over 1 h, followed by aqueous workup and column chromatography over silica gel 3,4-dihydrobenz[*f*]isoquinoline (**5**) is obtained in 25% to 33% yield.

Experimental Section

Unless otherwise noted, materials were obtained from commercial suppliers and were used without further purification. All reactions were conducted under an argon atmosphere. Melting points (Pyrex capillary) were measured on a Thomas-Hoover apparatus and are uncorrected. NMR spectra were determined on a Varian XL-300 spectrometer. Chemical shifts are expressed in parts per million downfield from internal tetramethylsilane. Significant ^1H NMR data are tabulated in the following order: number of protons, multiplicity (s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet), coupling constant(s) in hertz. EI mass spectra were obtained on a VG Model 7035 mass spectrometer and relevant data are tabulated as *m/z* (%RIC). Elemental analyses were performed by the Analytical Department, Merck Sharp and Dohme Research Laboratories, West Point, PA.

2-[(Ethoxycarbonyl)methyl]-3-carboxynaphthalene (7). Sodium metal (2.25 g. 97.4 mmol) was dissolved in 300 mL of absolute ethanol. To this solution were added ethyl acetoacetate (8.26 mL, 64.8 mmol), copper(I) bromide (4.64 g, 32.4 mmol), and 2-bromo-3-carboxynaphthalene (8.13 g, 32.4 mmol). This mixture was heated at reflux for 3 h, cooled to room temperature, and filtered through a Celite pad. The solvent was removed in vacuo and the residue was partitioned between 2 N HCl and chloroform. The layers were separated and the milky chloroform solution was diluted with a little methanol. This solution was dried ($MgSO_4$), filtered, and concentrated in vacuo. The crude product (9.0 g) was chromatographed on 250 g of silica gel by using 3:97 methanol–chloroform as eluant. There was obtained 4.19 g of **7** as a tan solid. An analytical sample was prepared by recrystallization from 9:1 hexanes–ethyl acetate: mp 156–157 °C; ^1H NMR ($CDCl_3$) δ 1.31 (3 H, t, J = 7.1), 4.21 (2 H, s), 4.22 (2 H, q, J = 7.1), 7.61 (2 H, m), 7.74 (1 H, s), 7.85 (1 H, d, J = 8.0), 7.97 (1 H, d, J = 8.0), 8.77 (1 H, s); MS, *m/z* (relative intensity) 258 (3.9), 212 (43.5), 168 (54.9), 140 (100). Anal. Calcd for $C_{15}H_{14}O_4$: C, 69.76, H, 5.46. Found: C, 70.07; H, 5.57.

Benzo[*g*]homophthalimide (8). Half-ester **7** (5.19 g, 20.1 mmol) and urea (30.0 g, 0.5 mol) were ground together in a mortar. This mixture was melted in a 150 °C oil bath for 45 min. The melt was cooled to room temperature and the solid was suspended in 50 mL of water. The product was collected by filtration and the filter cake was washed with water (3 × 100 mL). The crude product was dried in vacuo and chromatographed on 150 g of silica gel using chloroform as eluant. There was obtained 2.21 g of **8** as an off-white solid: mp 250–251 °C; ^1H NMR (DMSO-d_6) δ 4.19 (2 H, s), 7.63 (2 H, m), 7.87 (1 H, s), 7.89 (1 H, d, J = 7.5), 8.11 (1 H, d, J = 7.5), 8.16 (1 H, s), 8.74 (1 H, s); MS, *m/z* (relative intensity) 211 (81), 168 (55), 140 (100). An acceptable analysis was not obtained for this compound.

1,2,3,4-Tetrahydrobenz[*g*]isoquinoline (9). To a solution of imide **8** (1.10 g, 4.26 mmol) in THF (100 mL) was added borane–THF complex (20.5 mL of a 1.0 M solution in THF, 20.5 mmol). This solution was heated at reflux for 20 h. The cooled reaction mixture was treated with methanol (25 mL) and the solvents were removed in vacuo. The residue was heated with 2 N HCl (100 mL) at reflux for 3 h. The solution was cooled, made basic with concentrated NH_4OH, and extracted with chloroform. The chloroform extract was dried (Na_2SO_4), filtered, and concentrated in vacuo to give 0.77 g of **9** as yellow crystals. For analytical purposes the HCl salt was prepared from ethanolic HCl: mp (HCl salt) 290–295 °C; ^1H NMR ($CDCl_3$, free base) δ 1.85 (1 H, br s), 2.98 (2 H, t, J = 6.1), 3.20 (2 H, t, J = 6.1), 4.19 (2 H, s), 7.36 (2 H, m), 7.40 (1 H, s), 7.55 (1 H, s), 7.72 (2 H, m). Anal. Calcd for $C_{13}H_{13}N \cdot HCl$: C, 71.07; H, 6.42; N, 6.37. Found: C, 70.91; H, 6.30; N, 6.04.

3,4-Dihydrobenz[*g*]isoquinoline (2). A solution of 1,2,3,4-tetrahydrobenz[*g*]isoquinoline (**9**) (0.69 g, 3.77 mmol) in methanol (65 mL) was added to a solution of sodium carbonate (4.40 g, 41.5 mmol) and Fremy's salt (2.02 g, 7.53 mmol) in water (110 mL). This mixture was stirred at room temperature for 2 h. The reaction mixture was diluted with water (500 mL) and extracted with chloroform (2 × 100 mL). The combined chloroform extracts were dried (Na_2SO_4), filtered, and conentrated in vacuo. The crude product was chromatographed on 50 g of silica gel by using 3:97 methanol–ethyl acetate as eluant. There was obtained 0.61 g of

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2 as white crystals. For analytical purposes the HCl salt was prepared from ethanolic HCl: mp (HCl salt) 225–229 °C; ^{1}H NMR ($CDCl_3$, free base) δ 3.88 (2 H, t, J = 6.6), 3.87 (2 H, dt, J = 2.1, 6.6), 7.51 (2 H, m), 7.59 (1 H, s), 7.78 (1 H, s), 7.81 (1 H, d, J = 8.0), 7.90 (1 H, d,, J = 8.0), 8.56 (1 H, br s); MS, m/z (relative intensity) 181 (100), 152 (48), 89 (21). Anal. Calcd for $C_{13}H_{11}N$·HCl: C, 71.72; H, 5.56; N, 6.43. Found: C, 71.95; H, 5.72; N, 6.39.

3,4-Dihydrobenz[*f*]isoquinoline (5). To a three-necked 1-L round-bottomed flask with mechanical stirrer, reflux condenser, and addition funnel were added Celite (34 g), phosphorus pentoxide (68 g, 479 mmol), and benzene (360 mL). To this well stirred, refluxing mixture was added a solution of 1-(2-formamidoylethyl)naphthalene (18.15 g, 91 mmol) in chloroform (120 mL), dropwise over 1 h. The mixture was maintained at reflux for 24 h, cooled to room temperature, and diluted with water (1 L). This mixture was stirred for 4 h and was filtered through a pad of Celite. The filtrate was washed with chloroform, made basic with concentrated NH_4OH, and extracted with chloroform (3 × 250 mL). The combined chloroform extracts were dried (Na_2SO_4), filtered, and concentrated in vacuo. The crude product was chromatographed on 100 g of silica gel using ethyl acetate as eluant. There was obtained 5.60 g of **5** as the free base. For analytical purposes the HCl salt was prepared from ethanolic HCl: mp (HCl salt) 190–191 °C; ^{1}H NMR ($CDCl_3$, HCl salt) δ 3.65 (2 H, t, J = 8.8), 4.22 (2 H, t, J = 8.8), 7.73 (2 H, m), 7.92 (3 H, m), 8.13 (1 H, d, J = 7.8), 9.46 (1 H, s); ^{13}C NMR ($CDCl_3$, HCl salt) δ 21.2, 40.9, 121.1, 124.8, 127.1, 128.1, 129.1, 130.7, 137.3, 137.7, 165.6; MS, m/z (relative intensity) 181 (100), 152 (36), 139 (7), 84 (13), 76 (18). Anal. Calcd for $C_{13}H_{11}N$·HCl: C, 71.72; H, 5.56; N, 6.43. Found: C, 71.39; H, 5.89; N, 6.11.

Registry No. 2, 112576-38-2; 2·HCl, 112576-39-3; 4, 23950-49-4; 5, 23950-51-8; 5·HCl, 112576-40-6; 6, 20717-80-0; 7, 112576-41-7; 8, 112576-42-8; 9, 21628-46-6; 9·HCl, 112576-43-9; $AcCH_2CO_2Et$, 141-97-9; $(NH_2)_2CO$, 57-13-6.

An Attempted Synthesis of Biotin

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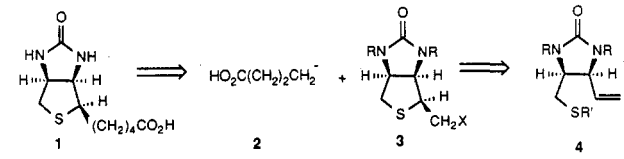
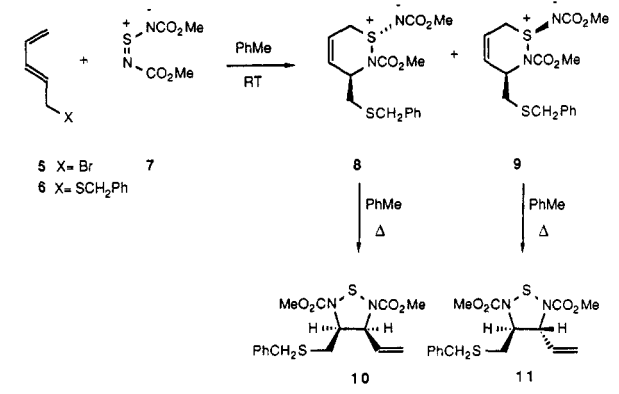
Renewed interest in the total synthesis of biotin (1)[1] has been spurred by recent findings[2] that emphasize the importance of this substance in the areas of nutrition and growth promotion. Despite efforts to develop new and efficient approaches to 1, the cofactor is still prepared commercially by the original Sternbach synthesis.[3] We have been attempting to effect a short, high yield, stereoselective total synthesis of biotin via the novel strategy outlined in Scheme I. It was our intention to prepare tetrahydrothiophene derivative 3, where X is an appropriate leaving group, and couple it with a synthetic equivalent for carbanion 2 to produce 1. We hoped that 3 could be synthesized from olefinic sulfide 4, which should

Scheme I



Scheme II



be available via our recently reported[4,5] methodology for stereoselective synthesis of unsaturated vicinal diamines. Clearly, in order for this route to be successful, the key cyclization of 4 to 3 must be achieved with the proper regiochemistry and stereochemistry at C-2.

The requisite erythro vicinal diamine derivative (cf. 4) was synthesized as outlined in Scheme II starting from the known (*E*)-pentadienyl bromide 5.[6] This compound upon treatment with the sodium salt of benzyl mercaptan was converted to sulfide 6 (97% yield). Diels–Alder cycloaddition of 6 with sulfur diimide 7 occurred in high yield in toluene at room temperature to produce a 7.7:1 mixture of epimeric dihydrothiazine imines 8 and 9. As anticipated from earlier work, no other regioisomeric adducts were detected.[5] Compounds 8 and 9 were not readily separable, so the mixture was used directly in the next step.

Heating the 7.7:1 mixture of adducts 8 and 9 in refluxing toluene induced a [2,3]-sigmatropic rearrangement, giving thiadiazolidines 10 and 11 in a 7.7:1 ratio in quantitative yield. We have previously shown that this type of rearrangement is stereospecific, with isomers having the stereochemistry shown in 8 affording 10 and 9 giving 11.[4,5] In fact, thermal rearrangement of a purified sample of major adduct 8 cleanly produced thiadiazolidine 10. The mixture of 10 and 11 could be readily separated at this stage by flash chromatography, and the desired major thiadiazolidine 10 was carried on.

Reduction of 10 with $NaBH_4$ gave the erythro dicarbamate 12 (91%). This compound was cyclized by using sodium hydride to urea 13 (89%).[4] Interestingly, the dibenzylated urea 14 could be prepared in a single step in 96% yield by a similar cyclization in the presence of benzyl bromide.

Treatment of olefinic sulfide 13 with bromine or NBS produced a new compound, which we believe is a cyclization product. However, the material was extremely insoluble and decomposed upon attempted chromatographic purification. On the other hand, sulfide 14 reacted cleanly

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