L-*N*-(Benzyloxycarbonyl)-3-(3-hydroxy-4-(benzyloxy)-phenyl)alanine Methyl Ester (17). Sodium borohydride (324 mg, 8.64 mmol, 1.5 equiv) was added to a solution of ketone **13** (2.70 g, 5.87 mmol) in dry methanol (27 mL) at 10 °C, and the reaction mixture was stirred for 2 h (25 °C). The reaction mixture was poured onto 5% aqueous HCl and was extracted with $CH_2Cl_2$ (3 × 50 mL). The organic extracts were washed with saturated aqueous sodium chloride, dried ($MgSO_4$), and concentrated in vacuo to afford the alcohol **15** as a colorless oil which was used directly in the following reaction. For **15**: $^1H$ NMR ($CDCl_3$, 200 MHz, ppm) 7.85 (d, 1 H, $J$ = 8 Hz, OH), 7.26 (br s, 10 H, two Ph), 6.80 (dd, 1 H, $J$ = 8, 3 Hz, C6-H), 6.68 (d, 1 H, $J$ = 8 Hz, C5-H), 6.62 (d, 1 H, $J$ = 3 Hz, C2-H), 5.23 (br d, 1 H, $J$ = 8 Hz, NH), 5.09 (s, 2 H, PhC$H_2$O), 5.06 (s, 2 H, PhC$H_2O_2$C), 4.92 (p, 1 H, $J$ = 8 Hz, ArC(OH)$H$CH$_3$), 4.61 (q, 1 H, $J$ = 8 Hz, CH$_2$C$H$NH), 3.73 (s, 3 H, OCH$_3$), 3.19 and 3.05 (two dd, 1 H each, $J$ = 16, 8 Hz, C$H$HCHNH and CH$H$CHNH), 1.51 (d, 3 H, $J$ = 8 Hz, ArCHC$H_3$).

A solution of alcohol **15** (2.72 g, 5.87 mmol) in 11.5 mL of THF was treated at 23 °C with 30% $H_2O_2$ (6.02 mL, 58.7 mmol, 10.0 equiv) and $p$-TsOH·$H_2O$ (345 mg, 1.75 mmol, 30 mol %). The reaction mixture was stirred at 23 °C (24 h), diluted with half-saturated $NaHCO_3$ (10 mL), and extracted with $Et_2O$ (3 × 15 mL). The combined organic extracts were dried ($MgSO_4$) and concentrated in vacuo. Flash chromatography ($SiO_2$, 5 × 25 cm, 20% EtOAc–hexane eluant) afforded **17** (1.53 g, 2.55 g theoretical yield, 60%) as a colorless oil: $[\alpha]^{22}_D$ −15.1° ($c$ 1.0, MeOH); $^1H$ NMR ($CDCl_3$, 200 MHz, ppm) 7.39 (br s, 5 H, Ph), 7.33 (br s, 5 H, Ph), 6.80 (d, 1 H, $J$ = 8.2 Hz, C5-H), 6.69 (d, 1 H, $J$ = 2 Hz, C2-H), 6.55 (dd, 1 H, $J$ = 8.2, 2 Hz, C6-H), 5.62 (br s, 1 H, OH), 5.23 (br d, 1 H, $J$ = 8 Hz, NH), 5.09 (s, 2 H, PhC$H_2$O), 5.06 (s, 2 H, PhC$H_2O_2$C), 4.61 (q, 1 H, $J$ = 8 Hz, CH$_2$C$H$NH), 3.72 (s, 3 H, OCH$_3$), 3.18 and 3.09 (two dd, 1 H each, $J$ = 16, 8 Hz, C$H$HCHNH and CH$H$CHNH); IR (neat) $\nu_{max}$ 3854, 3838, 3816, 3807, 3745, 3676, 3347, 2954, 1719, 1696, 1685, 1646, 1590, 1576, 1539, 1507, 1457, 1432, 1341, 1275, 1215, 1129, 1061, 739, 697 cm$^{-1}$; EIMS, $m/e$ (relative intensity) 435 ($M^+$, 4), 303 (5), 284 (4), 213 (7), 158 (9), 156 (31), 141 (9), 139 (33), 111 (11), 91 (base); HRMS, $m/e$ 436.1736 ($C_{25}H_{29}NO_6$ requires 436.1760). Chiral-phase HPLC analysis[19] revealed a 95:5 ratio of L/D-**17**; $t_R$ = 18 min/28 min, 2.0 mL/min, 10% 2-propanol–hexane.

L-*N*-(Benzyloxycarbonyl)-3-(3-hydroxy-4-(benzyloxy)-phenyl)-*N*-methylalanine Methyl Ester (18). Sodium borohydride (20 mg, 0.51 mmol, 1.5 equiv) was added to a solution of the ketone **14** (164 mg, 0.34 mmol) in 1.5 mL of MeOH at 25 °C and the reaction mixture was stirred at 25 °C (1 h). The reaction mixture was poured onto 5% aqueous HCl and was extracted with $CH_2Cl_2$ (3 × 2 mL). The organic extracts were dried ($MgSO_4$) and concentrated in vacuo to afford alcohol **16** as a colorless oil which was used without purification. For **16**: $^1H$ NMR ($CDCl_3$, 200 MHz, ppm) 7.85 (d, 1 H, $J$ = 8 Hz, OH), 7.26 (br s, 10 H, two Ph), 6.80 (dd, 1 H, $J$ = 8, 3 Hz, C6-H), 6.68 (d, 1 H, $J$ = 8 Hz, C5-H), 6.62 (d, 1 H, $J$ = 3 Hz, C2-H), 5.09 (s, 2 H, PhC$H_2$O), 5.06 (s, 2 H, PhC$H_2O_2$C), 4.92 (p, 1 H, $J$ = 8 Hz, ArC(OH)$H$CH$_3$), 4.61 (q, 1 H, $J$ = 8 Hz, CH$_2$C$H$N), 3.73 (s, 3 H, OCH$_3$), 3.19 and 3.05 (two dd, 1H each, $J$ = 16, 8 Hz, C$H$HCHN and CH$H$CHN), 2.82 (s, 3 H, NCH$_3$), 1.51 (d, 3 H, $J$ = 8 Hz, ArCHC$H_3$).

A solution of alcohol **16** (154 mg, 0.32 mmol) in 1 mL of THF at 23 °C was treated with 30% $H_2O_2$ (0.33 mL, 3.2 mmol, 10 equiv) and $p$-TsOH·$H_2O$ (19 mg, 0.10 mmol, 30 mol %). The reaction mixture was stirred at 23 °C (24 h), diluted with half-saturated $NaHCO_3$ (0.5 mL), and extracted with $Et_2O$ (3 × 1 mL). The combined organic extracts were dried ($MgSO_4$) and concentrated in vacuo. Flash chromatography ($SiO_2$, 1 × 25 cm, 20% EtOAc–hexane eluant) afforded **18** (88 mg, 143 mg theoretical yield, 61%) as a colorless oil: $[\alpha]^{22}_D$ −7.0° ($c$ 1.0, MeOH); $^1H$ NMR ($CDCl_3$, 200 MHz, ppm) 7.39 (br s, 5 H, Ph), 7.33 (br s, 5 H, Ph), 6.80 (d, $J$ = 8.2 Hz, C5-H), 6.69 (d, 1 H, $J$ = 2 Hz, C2-H), 6.55 (dd, 1 H, $J$ = 8.2, 2 Hz, C6-H), 5.62 (br s, 1 H, OH), 5.09 (s, 2 H, PhC$H_2$O), 5.06 (s, 2 H, PhC$H_2O_2$C), 4.61 (t, 1 H, $J$ = 8 Hz, CH$_2$C$H$N), 3.72 (s, 3 H, OCH$_3$), 3.17 and 3.07 (two dd, 1 H each, $J$ = 16, 8 Hz, C$H$HCHN and CH$H$CHN), 2.82 (s, 3 H, NCH$_3$); IR (neat) $\nu_{max}$ 3372, 3065, 3033, 2592, 1741, 1703, 1592, 1511, 1455, 1403, 1382, 1320, 1274, 1217, 1130, 1011, 914, 855, 795, 766, 740, 699 cm$^{-1}$; CIMS (isobutane), $m/e$ 450 ($M^+$ + H, base), 406 ($M^+$ + H − $CO_2$, 81); HRMS, $m/e$ 449.1839 ($C_{26}H_{27}NO_6$ requires 449.1838). Chiral-phase HPLC analysis[19] revealed a 95:5 ratio of L/D-**18**; $t_R$ 16 min/25 min, 2.0 mL/min, 10% 2-propanol–hexane.

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**Registry No.** **1**, 64755-14-2; **2**, 64725-24-2; **3**, 106283-67-4; **4**, 106235-26-1; **5**, 70840-66-3; **6**, 86849-13-0; **7**, 86229-97-2; **8**, 70840-66-3; **9**, 83858-82-6; **10**, 32404-28-7; **11**, 57085-32-2; **12**, 110774-03-3; **13**, 105205-69-4; **14**, 110774-04-4; **15**, 105205-68-3; **16**, 110774-05-5; **17**, 105229-41-2; **18**, 110774-06-6; H-Tyr-OH, 60-18-4; AcCl, 75-36-5; PhCH$_2$OCOCl, 501-53-1; PhCH$_2$Br, 100-39-0.

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## Angoluvarin, an Antimicrobial Dihydrochalcone from *Uvaria angolensis*

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The genus *Uvaria* is a member of the plant family Annonaceae and has been a rich and varied source of new natural products, several of which have interesting biological activity.[1] *Uvaria angolensis* has previously yielded dihydrochalcones, flavanones, and benzylated indole alkaloids.[2] An investigation of another active column fraction[2a] has resulted in the isolation of an antimicrobially active dihydrochalcone for which the name angoluvarian has been chosen. It represents the most complex of the active dihydrochalcones yet isolated.

Angoluvarin (**1**) has molecular formula $C_{30}H_{28}O_6$ as determined by mass spectroscopy and combustion analysis. The 60-MHz $^1H$ NMR (acetone-$d_6$) data showed the characteristic $A_2B_2$ pattern for dihydrochalcones, five aromatic protons as a broad singlet ($\delta$ 7.20), one aromatic proton at $\delta$ 6.15 as a singlet, seven additional aromatic protons as a complex multiplet ($\delta$ 6.5–7.1), seven protons as a broad singlet at $\delta$ 3.80 (1 OCH$_3$ and 2 ArCH$_2$Ar), and four $D_2O$ exchangeable signals at $\delta$ 14.70, 4.80 (2 H), and 4.50. The low resolution mass spectrum shows a fragment ion peak at $m/z$ 379 ($M^+$ − 105), consistent for an unsubstituted B ring. These data suggest that angoluvarian (**1**) is a dibenzylated dihydrochalcone methyl ether. The 15-MHz $^{13}C$ NMR (acetone-$d_6$) data further support this conclusion with key signals located at $\delta$ 56.0 (q), 46.3 (t), 35.4 (t), 31.5 (t), 22.9 (t). The signal resonating at $\delta$ 35.4 (t) seems characteristic of the C-30 benzylic carbon of uvarinol, a tribenzylated flavanone previously isolated from *Uvaria chamae*.[3] The upfield signals at $\delta_C$ 92.1 (d) and $\delta_H$ 6.15 (1 H, s) suggest that they must be located ortho to a methoxyl group and therefore angoluvarin is benzylated at C-3′ and not C-5′. On the basis of previous

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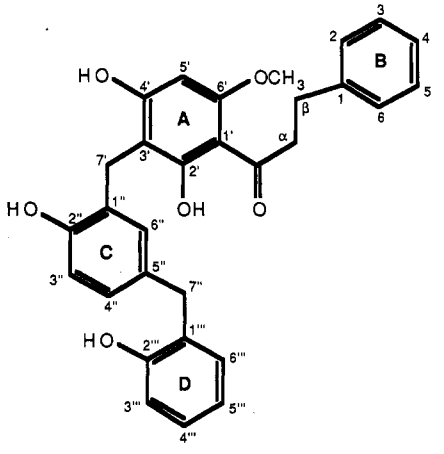
Table I. $^1H$ and $^{13}C$ NMR Shift Assignments for Angoluvarin (1)[a]

| carbon | $^1H$ multiplicity (J, Hz) | $^{13}C$[b] |
|---|---|---|
| 1 | | 141.4 (0), (128.2, 30.5), (2.93) |
| 2 | 7.26 m | 128.2 (1), (141.4), (2.93, 7.17) |
| 3 | 7.26 m | 128.2 (1), (125.8), (-) |
| 4 | 7.17 m | 125.8 (1), (128.2), (7.26) |
| 5 | 7.26 m | 128.2 (1), (125.8), (-) |
| 6 | 7.26 m | 128.2 (1), (141.4), (2.93, 7.17) |
| 1′ | | 104.3 (0), (203.3), (6.16, 14.5) |
| 2′ | | 164.3 (0), (-), (3.81, 14.5) |
| 3′ | | 106.1 (0), (162.7), (3.81, 6.16, 14.5) |
| 4′ | | 162.7 (0), (106.1), (3.81, 6.16) |
| 5′ | 6.16 s | 91.0 (1), (106.1, 161.1), (-) |
| 6′ | | 161.1 (0), (91.0), (6.16) |
| 7′ | 3.68 s | 21.9 (2), (-), (-) |
| 1″ | | 126.5 (0), (-), (3.81) |
| 2″ | | 152.6 (0), (114.5), (3.81, 6.70, 6.79) |
| 3″ | 6.75 d (8.0) | 114.5 (1), (152.6, 126.4), (-) |
| 4″ | 6.79 dd (8.0, 2.0) | 126.4 (1), (-), (3.80) |
| 5″ | | 131.0, (0), (-), (3.80) |
| 6″ | 6.70 d (2.0) | 129.0 (1), (-), (3.68, 3.80) |
| 7″ | 3.80 s | 34.5 (2), (-), (-) |
| 1‴ | | 127.9 (0), (154.7, 129.8), (3.80, 6.66, 6.82) |
| 2‴ | | 154.7 (0), (127.9, 114.9), (3.80, 6.99, 6.86) |
| 3‴ | 6.82 dd (8.5, 1.5) | 114.9 (1), (154.7, 126.6), (-) |
| 4‴ | 6.99 dt (8.5, 1.5) | 126.6 (1), (114.9, 118.7), (-) |
| 5‴ | 6.66 dt (8.5, 1.5) | 118.7 (1), (129.8, 126.6), (-) |
| 6‴ | 6.86 dd (8.5, 1.5) | 129.8 (1), (127.9, 118.7), (3.80) |
| $C_\alpha$ | 3.30 t (7.5) | 45.0 (2), (30.5, 203.7), (-) |
| $C_\beta$ | 2.93 t (7.5) | 30.5 (2), (45.0, 141.4), (-) |
| $OCH_3$ | 3.81 s | 55.6 (3), (-), (-) |
| CO | | 203.7 (0), (104.3, 45.0), (-) |
| 2′-OH | 14.38[c] | |
| 4′,2′,2″-OH | 9.33 s (1 H) and 10.0 (2 H)[c] | |

[a] Solvent $Me_2SO$-$d_6$. [b] Numbers in the first parentheses equal the number of attached hydrogens, numbers in the second parentheses represents the carbon chemical shift for which a correlation was present in the 2D-INADEQUATE experiment, numbers in the third parentheses represent chemcial shifts of protons for which long-range correlations were noted in the long-range heterocorrelation experiment. [c] Exchanges with $D_2O$.

studies[2b,3] the signal $\delta_C$ 22.9 (t) suggests that one of the benzyl groups is located at C-3′ between two hydroxyl groups (C-2′ and C-4′). On the basis of these data a highly tentative structure can be proposed for angoluvarin as shown by 1.




1

X-Ray diffraction studies to confirm the proposed structure were considered but suitable crystals could not be obtained after repeated attempts. The confirmation of the proposed structure was achieved by a detailed analysis of the high-field $^1H$ and $^{13}C$ NMR data.

The 300-MHz $^1H$ NMR data (Table I) reveal nearly every pattern as predicted by first-order analysis and are totally consistent with 1. The 75-MHz $^{13}C$ NMR data reveal 27 signals with an intense peak at 128.2 representing the two equivalent carbons for C-2,6 and C-3,5, thus accounting for all 30 carbon atoms. The APT experiment confirms the number of attached protons and the 1-bond heteronuclear correlation experiment correlates directly bonded protons to the corresponding carbons.

The long-range heteronuclear correlation data completely confirms the structure shown in 1. The methoxyl protons clearly correlate to the nonprotonated carbon-6′ while the $CH_2$ protons (H-7′) correlate to the nonprotonated carbons 2′, 4′, 2″, 1″, and 3′. The carbons 2′, 4′, and 3′ must be on the A ring since 4′ and 3′ show correlations to H-5′ (as do 1′ and 6′ also) and 2′, 3′, and 1′ correlate with the folded-in hydroxyl proton located at $\delta$ 14.5, which must be adjacent to the carbonyl. The $CH_2$ protons (H-7′) also correlate to the protonated carbon 6″, which must therefore be on the C ring along with C-1″ and C-2″.

C-2″ also shows strong three-bond correlations with H-6″ and H-4″, which must therefore be meta to the hydroxyl. A second pair of protons $CH_2$ (H-7‴) correlate to the protonated carbons 6‴, 6″, and 4″, which places C-6‴ on the D ring, and also correlate to the nonprotonated carbons 2‴, 5″, and 1‴. Carbon 2‴, which is on the D ring, correlates to H-4‴ and H-6‴, which requires placement of C-6‴ and C-4‴ as shown in 1. The C-13 chemical shifts of C-3″ and C-3‴ are consistent with those typical of aromatic protons ortho to the hydroxyl group in a phenolic ring structure, while C-5‴ exhibits a slightly larger chemical shift typical of an aromatic carbon para to a hydroxyl group. The long-range correlation data show that both H-5‴ and H-3‴ correlate with C-1‴, so they must be located on the D ring as shown, leaving C-3″ to be assigned

(3) Hufford, C. D.; Lasswell, W. L. *Lloydia* **1978**, *41*, 151.

to the position ortho to C-2″ in the C ring.

The $CH_2$ protons ($H_\beta$) correlate to C-1 through a two-bond coupling and to the two equivalent carbons (C-2 and C-6) through three-bond couplings. The carbons C-2 and C-6 show a correlation to the proton H-4 and reciprocally C-4 shows a correlation to the protons H-2 and H-6. Thus, the B ring is connected to the three-carbon chain at C-1 with C-2, C-6 ortho, C-3,5 meta, and C-4 para.

A carbon–carbon connectivity experiment (2D-INADEQUATE) was also performed and while not all of the correlations were observed, much of the structural information deduced from the long-range heterocorrelation was also independently confirmed. The following bonds were unequivocally confirmed: C=O,1′; C=O,α; α,β; β,1; 1,2-6; 3-5,4; 4′,5′; 6′,5′; 3′,4′; 2″,3″; 3″,4″; 2‴,1‴; 6‴,1‴; 6‴,5‴; 5‴,4‴; 4‴,3‴; and 2‴,3‴.

On the basis of the above arguments the structure for angoluvarin (1) has been established. All of the proton and carbon assignments are listed in Table I.

## Experimental Section

The one-dimensional 60-MHz $^1H$ NMR and the 15-MHz $^{13}C$ NMR data were obtained in acetone-$d_6$ on JEOL C-60 and JEOL FX-60 spectrometers, respectively. The multiplicities were confirmed by SFORD. The 300-MHz $^1H$ and 75-MHz $^{13}C$ NMR data (Table I) were run on a Varian XL-300 spectrometer.

The 2D heteronuclear one-bond and long-range chemical shift correlation data were obtained with the pulse sequence of Bax.[4] Both experiments were run with a spectral width of 5000 Hz in F2 (the carbon chemical shift axis) and 1400 Hz in F1 (the proton chemical shift axis), with the spectral windows centered around the protonated carbons and the corresponding protons, respectively. Some correlation peaks were lost, and others folded into the spectrum, but these disadvantages were more than offset by the improved digital resolution that could be maintained with the restricted spectral windows. A data acquisition time of 205 ms gave 2048 data points in F2. The number of increments was 128, and after Fourier transformation and zero-filling in F1 to 512 data points, a second Fourier transformation gave a data matrix of 2048 × 512. Contour plotting was used to identify the proton–carbon correlation peaks. Pulse sequence timing was determined by setting the value of $J_{CH}$, the direct coupling constant, to 140 Hz, and $J_{nCH}$, the long-range coupling constant, to 7 Hz.

Carbon–carbon connectivities were verified with the CCC2DQ 2D-INADEQUATE pulse sequence with quadrature detection in both dimensions as reported by Bax et al.[5] A spectral width of 14084.5 Hz in both F2 (the carbon chemical shift axis) and F1 (the double quantum frequency axis) was employed, with an acquisition time of 145 ms and 128 increments. Zero-filling to 512 data points in F1 gave a data matrix of 4096 × 512 data points to present the transformed 2D data. The high concentration of the sample (130 mg in 0.2 mL) in DMSO-$d_6$ resulted in sufficiently short $T1$'s for all carbons to optimize magnetization recovery with a sequence repetition time of 0.645 s. Acquisition of 2560 pulses per increment resulted in a total data acquisition of 59 h. Contour plotting revealed carbon–carbon connectivities as pairs of doublets with the same double quantum frequency appearing opposite the chemical shift positions of carbons joined by a chemical bond. A compromise value of $J_{CC}$ = 50 Hz was used to determine the pulse-sequencing timing in order to detect both single bonds and aromatic C–C bonds.

**Extraction and Chromatographic Separation.** The plant material used, the extraction procedures, and chromatographic separations have been reported previously.[2a,b] Angoluvarin was eluted in a fraction along with (+)-6,8-C-dimethylpinocembrin 6-methyl ether from the silicic acid column. Further chromatography ove alumina as described previously[2b] gave 50 mg of angoluvarin (elution with 5% MeOH–$CHCl_3$).

**Angoluvarin**: mp 154–156 °C; UV (MeOH) $\lambda_{max}$ 326 mm ($\epsilon$ 1.27 × $10^4$), 286 (9.97 × $10^4$), 250 (6.34 × $10^3$), and 218 (2.84 × $10^4$); IR (KBr) $\nu_{max}$ 3540, 3260, 1630, and 1600 $cm^{-1}$; MS, $m/z$ (relative intensity) 484 ($M^+$, 39), 379 ($M^+$ – 105, 15), 352 (22), 273 (17), 179 (100); $R_f$ 0.45 ($CHCl_3$–EtOH, 19:1). Anal. Calcd for $C_{30}H_{28}O_6$: C, 74.36; H, 5.82. Found: C, 74.04; H, 6.01.

**Antimicrobial Activity.** The antimicrobial assay was performed as previously described.[6] Angoluvarin had MIC values of 0.78, 1.56, and 3.12 μg/mL against *Bacillus subtilis*, *Staphylococcus aureus*, and *Mycobacterium smegmatis*, respectively.

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# Communications

## Asymmetric Oxidation of Chiral Enolates in the Preparation of Acyclic Tertiary α-Hydroxy Amides in High Optical Purity

*Summary:* Asymmetric oxidation of chiral acyclic, tetrasubstituted enolate **4c** with oxaziridines (+)-**1**/(−)-**2** in the presence and absence of HMPA affords optically active tertiary α-hydroxy amide **5c** in high optical purity (88–91% de). The application of (+)-**1**/(−)-**2** as "chiral probes" of enolate–electrophile reaction mechanisms is proposed.

*Sir:* Optically active acyclic tertiary α-hydroxy carbonyl compounds are valuable intermediates in the enantioselective synthesis of complex natural products such as insect pheromones[1] and antibiotics.[2] Generally these compounds are prepared by addition of organometallic reagents to optically active α-keto amides,[3] esters,[1b] oxazolines,[4] 1,3-oxathianes[1c,2,5] or the alkylation of α-keto amide dianions.[6] Recently we described methodology for the direct introduction of the hydroxy group adjacent to the carbonyl group via asymmetric enolate oxidation using the readily available camphorylsulfonyl oxaziridines (+)-**1** and (−)-**2** (50–95% ee).[7,8] High diastereofacial selectivity has also been reported for the oxidation of chiral enolates using 2-(phenylsulfonyl)-3-phenyloxaziridine (**3**),[9,10] MoOPH[11]

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