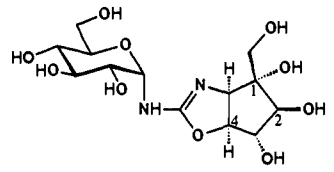
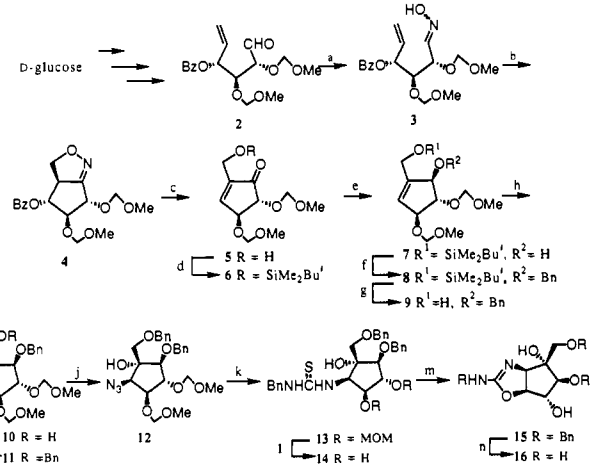


Trehazolin (1)

Figure 1.

Scheme I[a]

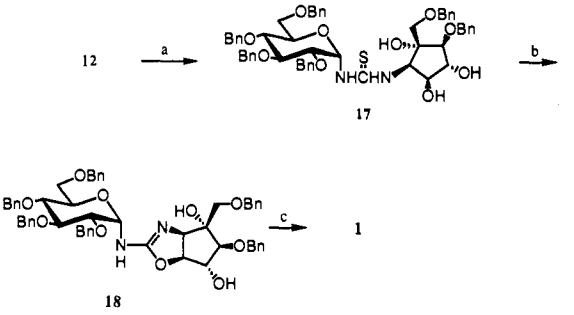


[a] (a) 10 equiv of NH_2OH·HCl, 10 equiv of Na_2CO_3, Et_2O–H_2O, 25 °C, 8 h, 74%. (b) Aqueous 5% NaOCl, catalytic Et_3N, CH_2Cl_2–H_2O, 0 °C, 40 min, 66%. (c) H_2, Raney Ni, $B(OH)_3$, 1,4-dioxane–MeOH–H_2O, 25 °C, 5 h, 72%. (d) 1.5 equiv of *tert*-$BuMe_2SiCl$, 1.5 equiv of imidazole, DMF, 25 °C, 16 h, 88%. (e) 1.5 equiv of $NaBH_4$, 1.5 equiv of $CeCl_3$·$7H_2O$, MeOH, 0 °C, 2 h, 53%. (f) 1.5 equiv of BnBr, 1.5 equiv of NaH, DMF, 0 °C, 1 h. (g) 1.5 equiv of *n*-Bu_4NF, THF, 0 °C, 1 h, 58% from **7**. (h) 1.4 equiv of $Ti(OPr^i)_4$, 1.5 equiv of diisopropyl L-tartrate, 2.0 equiv of *t*-BuOOH, CH_2Cl_2, −20 °C, 5 h, 94%. (i) 1.5 equiv of BnBr, 1.5 equiv of NaH, DMF, room temperature, 2 h, 98%. (j) 12 equiv of NaN_3, 12 equiv of NH_4Cl, DMF–ethylene glycol, 125 °C, 48 h, 78%. (k) 4.0 equiv of $LiAlH_4$, Et_2O, 0 °C, 4 h, then 1.5 equiv of BnNCS, THF, room temperature, 3 h, 83%. (l) 0.5 M aqueous HCl, 1,4-dioxane, 60 °C, 24 h, 74%. (m) 1.2 equiv of 2-chloro-3-ethylbenzoxazolium tetrafluoroborate, MeCN, 0 °C, 1 h, then quenched with 2.4 equiv of Et_3N, 0 °C, 3 h, 82%. (n) H_2, $Pd(OH)_2$–C, MeOH, 60 °C, 30 min, 71%.

D-tartrate instead of L-tartrate gave a diastereomer of the epoxide **10** in 77% yield. The configuration of these diastereomers followed Sharpless' epoxidation rule.[9]

After benzylation of **10** with benzyl bromide–NaH in DMF, treatment of the benzylated product **11** with sodium azide,[10] ammonium chloride, and ethylene glycol in dimethylformamide gave azide **12**. Reduction of the azide group of **12** with lithium aluminum hydride in ether and treatment of the resulting amine with benzyl isothiocyanate gave thiourea **13**. Deprotection of two methoxymethyl groups of **13** by 0.5 M aqueous hydrogen chloride in 1,4-dioxane afforded **14**. Treatment of **14** with 2-chloro-3-ethylbenzoxazolium tetrafluoroborate[11] gave aminooxazoline **15** through an intermediate carbodiimide. The benzyl groups of **15** were removed with hydrogen using palladium hydroxide on carbon (Pearlman's catalyst) in methanol to give **16** ($[\alpha]^{25}_D$ +14.4° (*c* 0.32, H_2O)), which was identical with natural trehazolin aglycon ($[\alpha]^{25}_D$ +13.5° (*c* 0.74, H_2O))[12] in all respects. The absolute configuration of natural trehazolin aglycon was thus determined as [1*R*-(1α,2β,3α,4β,5β)].

Scheme II[a]



[a] (a) 4.0 equiv of $LiAlH_4$, Et_2O, 0 °C, 4 h, 5% HCl–MeOH, 60 °C, 5 h, and then 1.0 equiv of 2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranosyl isothiocyanate, 1.5 equiv of Et_3N, room temperature, 18 h, 69%. (b) 1.7 equiv of 2-chloro-3-ethylbenzoxazolium tetrafluoroborate, MeCN, 0 °C, 1 h, then quenched with 3.2 equiv of Et_3N, 0 °C, 1 h, 68%. (c) H_2, $Pd(OH)_2$–C, MeOH, 60 °C, 30 min, 44%.

Finally, we synthesized trehazolin as follows. Treatment of the amine obtained from **12** with 5% methanolic hydrogen chloride at 60 °C removed the two methoxymethyl groups, and then treatment of the resulting triol amine hydrochloride with 2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranosyl isothiocyanate[13] and triethylamine gave an α-D-glucopyranosylthiourea derivative **17**.[14] Cyclization of **17** with 2-chloro-3-ethylbenzoxazolium tetrafluoroborate[11] and triethylamine in acetonitrile gave hexabenzyltrehazolin **18**, which was hydrogenolized to trehazolin (**1**) using Pearlman's catalyst. The synthetic trehazolin (**1**, $[\alpha]^{30}_D$ +112.7° (*c* 0.59, H_2O)) was identical with natural trehazolin ($[\alpha]^{25}_D$ +99.5° (*c* 0.44, H_2O))[1] in all respects, including inhibition activity toward both silkworm and porcine trehalases.[1]

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(14) If deprotection of the two methoxymethyl groups of the resulting amine was done in the final step after the formation of the thiourea, a 1:1 mixture of **18** and its β-anomer was obtained.

Biosynthesis of Dehydrorabelomycin and PD 116740: Prearomatic Deoxygenation as Evidence for Different Polyketide Synthases in the Formation of Benz[*a*]anthraquinones

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During the past decade the structural diversity of known naturally occurring benz[*a*]anthraquinones has expanded, causing them to become a major class of polyketide metabolites.[1] The biosynthesis of three members has been reported: two are derived from the predictable folding of a decaketide precursor,[2-5] while a third may be derived by rearrangement of a linear intermediate.[8] Many benzanthraquinones possess a hydroxyl at C-6 (derived from C-1 of acetate, e.g., dehydrorabelomycin (**1**)[6,9]), while a nearly

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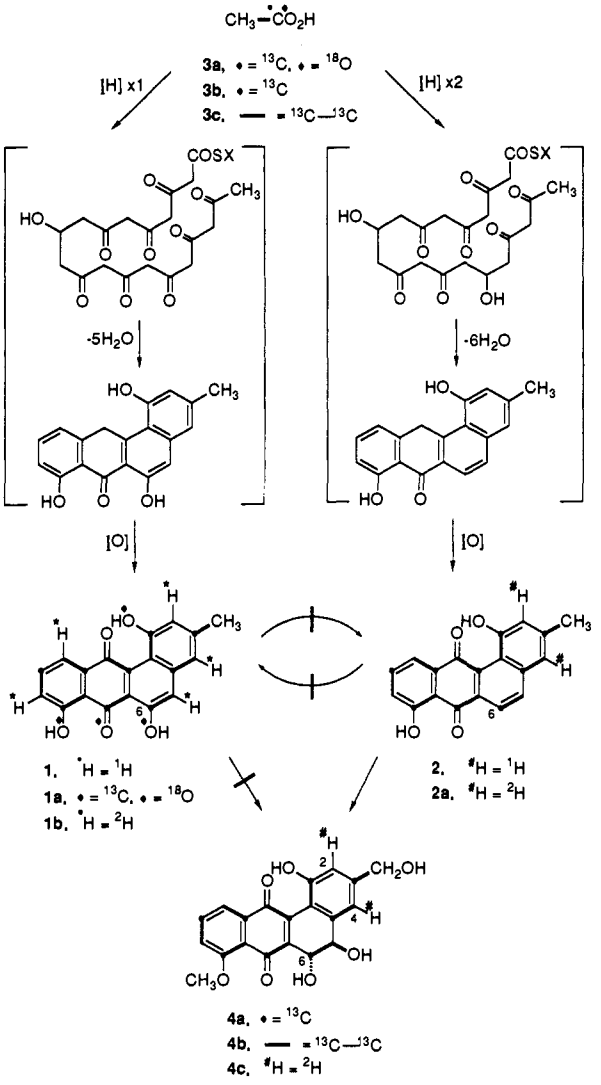
Scheme I



equal number lack oxygen at this site (e.g., tetrangulol (**2**)[10,11]). We now report data on a member of each subset that indicate that neither subset is the precursor of the other; rather, they are derived from different prearomatic intermediates.

We have previously reported the isolation of **1** from *Streptomyces murayamaensis* and the incorporation of **1** into kinamycin D by the same organism.[6] In order to determine the origin of the C-6 oxygen (C-6 is lost during the conversion of **1** into the kinamycins[7]), sodium [1-^{13}C,$^{18}O_2$]acetate (497 mg, **3a**) was fed to five 200-mL fermentations, and labeled **1a** (3.6 mg) was subsequently isolated and converted to its triacetate. ^{13}C NMR analysis revealed the expected enhancements at nine carbons, and relative to the specific enrichment at C-1, C-6, C-7, and C-8, the retention of ^{18}O at these sites was 84, 84, 58, and 82%, respectively. The enrichment at the C-6 hydroxyl clearly establishes its origin from an acyclic polyketide intermediate which had retained the acetate carbonyl at this site. The lower level of ^{18}O retention at C-7 presumably reflects exchange of the quinone carbonyl with the medium.

We next investigated the biosynthesis of the benz[*a*]anthraquinone antibiotic PD 116470 (**4**), produced by *Streptomyces* WP 4669.[12] Compound **4** was readily purified following the literature procedure. Through 1D and 2D NMR experiments we have confirmed the structure of **4** and obtained the complete ^{13}C NMR assignments. Sodium [1-^{13}C]acetate (480 mg total) (**3b**) was fed to three 330-mL broths in thirds at 20, 30, and 40 h after inoculation, and subsequent workup yielded 8.1 mg of **4a**. The ^{13}C NMR spectrum (100.6 MHz, MeOH-d_4) indicated that nine carbon atoms were enriched, as indicated on the structure. Sodium [1,2-$^{13}C_2$]acetate (400 mg) (**3c**) was fed next in order to establish the nature of the biosynthetic backbone. This yielded 16.4 mg of pure **4b**. The ^{13}C NMR spectrum of **4b** showed 18 resonances with doublets flanking the natural abundance singlets, indicating nine intact precursor acetate units and one lone enriched singlet, while the 2D INADEQUATE spectrum confirmed the expected pairings of the doublets. The labeling pattern revealed that **4** is derived by a simple folding and condensation of a decaketide to the angular benz[*a*]anthraquinone skeleton, as shown in Scheme I.

[2,4,5,9,11-$^2H_5$]-**1b**[6] and [2,4-$^2H_2$]-**2a**[13] were next tested as intermediates in the biosynthesis of **4**. Samples of **1b** and **2a** (20 mg in 2 mL of DMSO) were fed to separate fermentations using the same protocol as above. Purified **4** obtained from the **1b** feeding showed no enrichment by ^{2}H NMR spectroscopy. No remaining **1b** could be detected in the broth, indicating that it had been consumed. Workup of the **2a** feeding afforded 15 mg of **4c**, and 8.0 mg of **2a** was recovered unchanged. ^{2}H NMR analysis (61.4 MHz, methanol for chemical shift reference and deuterium quantitation) of **4c** clearly showed resonances at δ 6.90 and 6.95, corresponding to H-2 and H-4, respectively (average enrichment 1.38%).

In the biosynthesis of dehydrorabelomycin (**1**), the C-6 hydroxyl oxygen is retained from the original acetate precursor. Although aromatic hydroxylation is a well-known metabolic reaction, tetrangulol (**2**) is clearly not a precursor to **1**. Tetrangulol, rather than **1**, has now been shown to be the key intermediate in the biosynthesis of **4**. Recently postaromatic deoxygenation of a polyketide metabolite has been demonstrated,[15] but the results reported here show that such a process does not occur in the biosynthesis of **4**. Having undergone only oxidation at C-12 and decarboxylation at C-2, **1** and **2** are the two simplest benz[*a*]anthraquinones. Therefore, C-6 deoxygenation must occur at a prearomatic stage.[16] Since polyketide biosynthesis has been shown to be a "processive" metabolism,[17-19] the cumulative results indicate that **1** and **2** are generated from different polyketide synthases. Thus, there are two separate pathways, and **1** and **2** can now be considered the "parents" of two different large subfamilies of metabolites. As part of a program to explore the subsequent modifications of these compounds, the mechanism of the formation of **4** from **2** will be described in due course.

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through grants from the National Science Foundation (CHE-8216190) and from the M. J. Murdock Charitable Trust to Oregon State University, and the Bruker AC 300 spectrometer was purchased in part through grants from the Public Health Service Division of Research Resources (RR04039-01) and the National Science Foundation (CHE-8712343) to Oregon State University.

Synthesis and Structure of the First Uranium(VI) Organometallic Complex

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The chemistry of complexes of the d-transition elements containing organoimido functional groups constitutes a rapidly expanding area of research.[1] Examples of molecules containing multiple terminal imido functional groups now exist for representative elements from groups 5–8.[1,2] In contrast, f-metal terminal organoimido complexes are still relatively rare,[3] and examples of single actinide metal sites coordinated by greater than one imido ligand are unknown. On the basis of the stability of uranyl ion $(UO_2)^{2+}$, there appears to be no reason why the isoelectronic bis(organoimido) species should not be accessible.[4] Development of this chemistry has been hampered by the lack of synthetic routes to high-valent (V, VI) uranium complexes and by the relatively small number of ligands which have been demonstrated to stabilize these oxidation states.[3-5] Here we describe the synthesis and structural characterization of the first bis(organoimido) complex of uranium(VI). This species also represents the first complex of uranium(VI) supporting metal–carbon bonding in either a σ- or π-fashion.

As part of our studies of preparative routes to uranium organoimido complexes,[3a] we are examining generation of uranium imido functionalities by abstraction of an amido hydrogen by a leaving hydrocarbyl or amido ligand.[6] The room temperature reaction of red-orange $(C_5Me_5)_2U(CH_3)Cl$[7] with 1 equiv of $LiNHC_6H_5$ in diethyl ether for 12 h in the presence of 1 equiv of TMEDA results in the slow evolution of methane and the formation of a brown-orange solution. Following removal of the diethyl ether under reduced pressure, the residue is extracted with toluene, the extract is filtered through Celite, and the solvent is removed in vacuo. Treatment of the resulting red-brown oil with cold hexane yields $[Li(TMEDA)](C_5Me_5)_2U(NC_6H_5)Cl$ (**1**)[8] as a brown-orange powder in 75% yield (eq 1).[9]

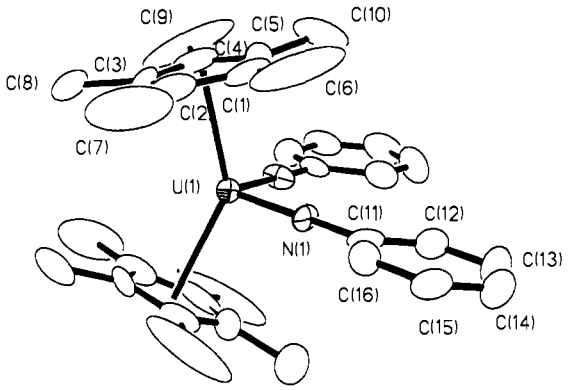

Figure 1. ORTEP drawing of $Cp^*_2U(NC_6H_5)_2$ (**2**), with atoms shown as 50% probability ellipsoids. Selected distances (Å) and angles (deg) for **2**: U(1)–N(1) = 1.952 (7), U(1)–N(1)–C(11) = 177.8 (6), N(1)–U(1)–N(1′) = 98.7 (4), Cp*(centroid)–U(1)–Cp* = 141.9.

$$(C_5Me_5)_2U(CH_3)Cl + 1LiNHC_6H_5 + TMEDA \xrightarrow[-CH_4]{(C_2H_5)_2O} [Li(TMEDA)](C_5Me_5)_2U(NC_6H_5)Cl \quad (1)$$
$$\mathbf{1}$$

The addition of 1 equiv of phenyl azide to a stirred solution of **1** in diethyl ether at room temperature results in a rapid color change from brown-orange to black-brown with the evolution of nitrogen.[3c] After 12 h the solvent is removed, yielding $(C_5Me_5)_2U(NC_6H_5)_2$ (**2**) as a black-green powder (85% yield) upon washing with cold hexane (eq 2).[10] Compound **2** is soluble in ether and aromatic solvents and exhibits only modest air and moisture sensitivity.

$$[Li(TMEDA)](C_5Me_5)_2U(NC_6H_5)Cl + 1N_3C_6H_5 \xrightarrow[-N_2]{(C_2H_5)_2O} (C_5Me_5)_2U(NC_6H_5)_2 \quad (2)$$
$$\mathbf{2}$$

Compound **2** may be more directly prepared in high yield (80%) by the addition of 1-lithio-1,2-diphenylhydrazine to a stirred diethyl ether solution of $(C_5Me_5)_2U(CH_3)Cl$ at room temperature (eq 3). Evolution of methane is vigorous, and the solution color changes from the deep maroon of $(C_5Me_5)_2U(CH_3)Cl$ to the black-brown of **2** within ca. 15 min. The intermediacy of a U(IV) η^2-1,2-diphenylhydrazido(2−) species may be postulated, which subsequently undergoes N–N bond cleavage to form **2**. Other

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(9) $[Li(TMEDA)](C_5Me_5)_2U(NC_6H_5)Cl$ (**1**): ^1H NMR (250 MHz, C_6D_6, 25 °C) δ 39.55 (s, $\nu_{1/2}$ = 31 Hz, 2 H, meta), 30.45 (s, $\nu_{1/2}$ = 31 Hz, 2 H, ortho), 13.79 (s, $\nu_{1/2}$ = 31 Hz, 1 H, para), 4.82 (br, $\nu_{1/2}$ = 78 Hz, 4 H, $Me_2NCH_2CH_2NMe_2$), 3.29 (br, $\nu_{1/2}$ = 250 Hz, 12 H, $Me_2NCH_2CH_2NMe_2$), −1.27 (s, $\nu_{1/2}$ = 15 Hz, 30 H, C_5Me_5); IR (Nujol mull) 1288 (s), 1272 (vs), 1264 (vs), 1224 (m), 1188 (w), 1160 (s), 1148 (w), 1120 (m), 1092 (w), 1069 (w), 1064 (m), 1031 (m), 1016 (m), 1010 (m), 992 (s), 984 (w), 977 (w), 952 (m), 900 (s), 876 (m), 828 (w), 792 (s), 783 (w), 764 (s), 724 (m), 692 (s), 672 (w), 628 (s), 608 (m), 592 (w), 584 (m), 492 (m). Anal. Calcd for $C_{32}H_{51}N_3LiClU$: C, 50.68; H, 6.79; N, 5.54. Found: C, 51.06; H, 6.69; N, 5.17.

(10) $(C_5Me_5)_2U(NC_6H_5)_2$ (**2**): ^1H NMR (250 MHz, C_6D_6, 25 °C) δ 9.26 (t, 4 H, J_{HH} = 7.75 Hz, meta), 4.16 (s, 30 H, C_5Me_5), 2.82 (d, 4 H, J_{HH} = 7.46 Hz, ortho), 0.72 (t, 2 H, J_{HH} = 7.35 Hz, para); ^{13}C NMR (62.9 MHz, C_6D_6, 25 °C): δ 181.2 (C_{phenyl}), 143.4 (C_{phenyl}), 136.5 (C_{ipso}/C_{ring}), 110.8 (C_{ipso}/C_{ring}), 109.7 (C_{phenyl}), 7.7 (C_5Me_5); IR (Nujol mull) 1273 (s), 1266 (s), 1252 (m), 1210 (vw), 1160 (m), 1142 (w), 1062 (m), 1014 (m), 992 (m), 905 (s), 889 (m), 860 (w), 819 (w), 756 (vs), 686 (vs), 601 (vw), 527 (s). Anal. Calcd for $C_{32}H_{40}N_2U$: C, 55.65; H, 5.84; N, 4.06. Found: C, 55.27; H, 6.06; N, 3.74.

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