J. Am. Chem. Soc. **1983**, *105*, 1401-1403

1 mM FeTPP(Cl) and O_2 (2 mol/mol of FeTPP(Cl)) and characterized by visible and ^1H NMR spectra superimposable on those of an authentic sample.[21] Complex **2′** is much more stable toward O_2 ($t_{1/2}$ in aerated $C_6H_6 \simeq 12$ h[21c]) than **2**. On the contrary, the diazene complex **1′**, $Fe^{II}TPP(C_6H_5N{=}NH)_2$, is much less stable than **1**; it has been only observed by visible spectroscopy (λ 426 nm, 529, 561) upon reaction of $C_6H_5N{=}NH$ with FeTPP in anaerobic conditions but could not be isolated so far because of its fast transformation into **2′** in the presence of O_2 traces. These data explain why **2′** is the main complex derived from the binding of an oxidized product of $C_6H_5NHNH_2$ to iron, observed during FeTPP and O_2-dependent oxidation of $C_6H_5NHNH_2$.

The aforementioned results establish the formation of two Fe-$RNHNH_2$-derived ligand complexes upon reaction of $RNHNH_2$ hydrazines or the corresponding diazenes with an iron porphyrin and O_2 (Scheme I): an Fe(II)-diazene and a σ-Fe(III)-R complex. They show, at least when R = CH_3, that the σ-Fe(III)-R complex can be formed by a O_2-dependent oxidation of the diazene Fe(II)-RN=NH complex. There are great similarities between these results and those concerning reactions of hemoglobin (Hb) or myoglobin (Mb) with $RNHNH_2$ (R = CH_3 or C_6H_5) and O_2:[10] when R = CH_3, two iron complexes are formed, the first one, a hemoprotein Fe(II)-$CH_3N{=}NH$ complex being further oxidized by 1 equiv of $Fe(CN)_6K_3$ (or by O_2) to give another complex, B, of unknown structure. When R = C_6H_5, the Fe(II)-$C_6H_5N{=}NH$ complex has never been observed, the only entity formed being a complex spectrally similar to B.[27] This paper describes the first isolation of a porphyriniron(II)-alkyldiazene complex[22] and points to the great instability of Fe(II)-$C_6H_5N{=}NH$ complexes, thus strongly supporting the existence of Mb- (or Hb-) Fe(II)-$CH_3N{=}NH$ complexes[10] and explaining the failure to observe Mb-Fe(II)-$C_6H_5N{=}NH$ complexes.[9a,10,23] The diazene ligands are isoelectronic with their oxygen-containing analogues, nitrosoalkanes[15] and O_2, and it is noteworthy that, as their diazene analogues, nitrosoarene-iron porphyrin complexes are considerably less stable than nitrosoalkane-iron complexes.[15c] This paper also reports for the first time the formation of σ-Fe(III)-R complexes upon reaction of $RNHNH_2$ or RN=NH with iron porphyrins, indicating a possible $HbFe^{III}$-R (R = CH_3 or C_6H_5) structure for complexes B.[27] However, one cannot exclude presently the previously proposed[9] $HbFe^{III}$-N=NR structure for these complexes. In that respect, it is noteworthy that hydrazines $RNHNH_2$ react with Co and Fe chelates to give Co(III)-R and Fe(III)-R σ complexes,[24] whereas $C_6H_5NHNH_2$ reacts with molybdenum porphyrins to give Mo-N=NC_6H_5 complexes.[25]

Finally, very recent preliminary results are in favor of the formation of similar iron complexes during the cytochrome P-450 dependent oxidation of CH_3NHNH_2[26] and $C_6H_5NHNH_2$.[3,26]

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(21) (a) Reed, C. A.; Mashiko, T.; Bentley, S. P.; Kastner, M. E.; Scheidt, W. R.; Spartalian, K.; Lang, G. *J. Am. Chem. Soc.* **1979**, *101*, 2948. (b) The authentic sample was prepared by reaction of FeTPP(Cl) with C_6H_5MgBr.[21c] The ^1H NMR spectrum of **2′** (porphyrin signals δ −17.3, 3.0, 4.7, 4.9, 5.1, 6.0; phenyl ligand δ −25.55 (*p*-H), 13.4 (*m*-H) (the ortho hydrogens could not be detected because of their proximity to the paramagnetic center[18])) was found superimposable on that of the authentic sample. (c) Mansuy, D.; Battioni, J.-P.; Dupré, D.; Sartori, E.; Chottard, G. *J. Am. Chem. Soc.* **1982**, *104*, 6159-6161.

(22) Diimide, NH=NH, complexes of iron(II) porphyrins and hemoglobin-iron(II) have been described: Hanstein, W. G.; Lett, J. B.; McKenna, C. E.; Traylor, T. G. *Proc. Natl. Acad. Sci. U.S.A.* **1967**, *58*, 1314-1316.

(23) Huang, P. K. C.; Kosower, E. M. *Biochim. Biophys. Acta* **1968**, *165*, 483-489.

(24) Goedken, V. L.; Peng, S. M.; Park, Y. *J. Am. Chem. Soc.* **1974**, *96*, 284-285.

(25) Colin, J.; Butler, G.; Weiss, R. *Inorg. Chem.* **1980**, *19*, 3828-3833.

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(27) It was shown that an aerobic acidic treatment of this complex B′ formed upon reaction of Hb with $C_6H_5NHNH_2$ leads to *N*-phenylprotoporphyrin IX.[6d,e] Since recent results showed that *N*-phenylporphyrins are formed upon similar treatment of σ-(porphyrins)Fe^{III}-C_6H_5 complexes,[28a,21c] it is tempting to propose a σ-Fe^{III}-C_6H_5 structure for the Hb complex B′.[28b]

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Organolanthanide Hydride Chemistry. 3. Reactivity of Low-Valent Samarium with Unsaturated Hydrocarbons Leading to a Structurally Characterized Samarium Hydride Complex[1]

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Our general investigation of low-valent lanthanide chemistry[3-10] has shown that the zerovalent metals react with neutral unsaturated hydrocarbons[5-10] to form a variety of new classes of organolanthanides including some that display catalytic activity.[6,7] This low-valent approach also provided the first soluble divalent organosamarium complex, $(C_5Me_5)_2Sm(THF)_2$ (I) a complex that functions as a catalyst precursor for the catalytic hydrogenation of alkynes.[10] Since I was a crystallographically characterized low-valent complex that also had catalytic chemistry, it was an ideal candidate for the study of low-valent lanthanide reactivity. We report here the reaction of I with internal alkynes to form a new class of organolanthanide complexes, the enediyls, which can be converted into the first organosamarium hydride complex, a molecule that represents a new crystallographically characterized class of organolanthanide hydrides.[11,12]

Addition of pentane to an equimolar mixture of the purple I and $C_6H_5C{\equiv}CC_6H_5$ in an inert-atmosphere glovebox immediately generates an intensely colored black solution. Removal of solvent after stirring overnight yields a black material containing unreacted alkyne by ^1H NMR spectroscopy. A pentane solution of this material precipitates the excess $C_6H_5C{\equiv}CC_6H_5$ at −78 °C, leaving a black mother liquor that is pure by ^1H NMR spectroscopy. Removal of solvent from the mother liquor leaves a black glassy material, II, which by complete elemental analysis has the formula $[(C_5Me_5)_2SmCC_6H_5]_n$ (yield >95%). The ^1H NMR and IR spectra[13] are consistent with an enediyl structure, $[(C_5Me_5)_2Sm](C_6H_5)C{=}C(C_6H_5)[Sm(C_5Me_5)_2]$, and a trans configuration is likely on the basis of steric considerations.[14]

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(1) Presented in part at the 39th Southwest and 6th Rocky Mountain Combined Regional Meeting of the American Chemical Society, El Paso, TX, Dec 1982, Abstr. 194.

(2) (a) Camille and Henry Dreyfus Teacher-Scholar. (b) University of Chicago; current address University of California, Irvine, CA 92717. (c) University of Alabama.

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(13) ^1H NMR (C_6D_6) δ 1.74 (s, $C_5(CH_3)_5$). −1.26 (br s, C_6H_5), −10.15 (br s, C_6H_5) (a third broad C_6H_5 signal resolvable from the other resonances in the spectrum was not observed); ^{13}C NMR (C_6D_6) δ 123.2 (C_6H_5). 121.6 (C_6H_5), 119.4 ($\mathit{C}_5(CH_3)_5$), 115.3 (C_6H_5) 104.1 ($={\mathit{C}}C_6H_5$) 19.8 ($C_5(\mathit{C}H_3)_5$); IR (Nujol) 3770 (w), 1670 (w), 1580 (s), 1495 (sh), 1300 (m), 1200 (w), 1160 (m), 1070 (w), 1020 (m), 975 (m), 955 (m), 750 (s), 720 (d, s), 690 (d, s), 625 (w) cm^{-1}.

(14) Lithium reacts with $C_6H_5C{\equiv}CC_6H_5$ to form the cis dianion on the basis of hydrolytic analysis. Sodium reacts with this alkyne to form the trans dianion: House, H. O. "Modern Synthetic Reactions"; W. A. Benjamin: Menlo Park, CA, 1972; pp 205-209 and references therein.

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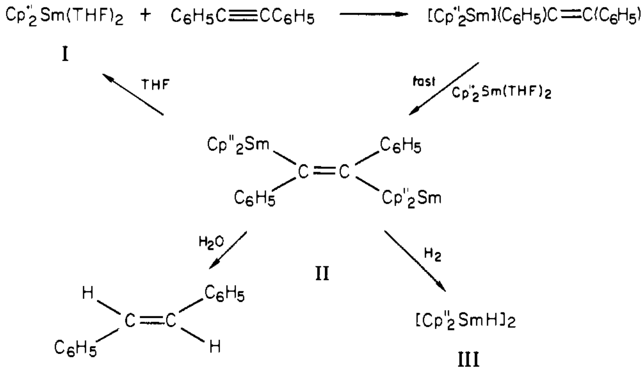
Scheme I



Cp'' = $C_5(CH_3)_5$

Hydrolysis generates pure *trans*-stilbene.

The formation of II can be rationalized (Scheme I) by initial formation of the radical, $[(C_5Me_5)_2Sm](C_6H_5)C{=}C(C_6H_5)$, which is rapidly reduced by a second equivalent of the soluble I before dimerization can occur.[15] Sm(II) is known to rapidly reduce radicals to anions,[16,17] and it has previously been shown that diarylalkynes can be reduced to dianions under special conditions at low temperature.[14] Both the magnetic susceptibility and the near-infrared spectrum of II are characteristic of Sm(III).[18] However, the visible spectrum of II differs from those of previously reported Sm(III) organometallic complexes, which are usually yellow or orange, in that it contains an intense charge-transfer-like absorption starting at 1050 nm, which is responsible for the black color.[19]

Since Sm(II) is the most reactive of the accessible divalent lanthanides,[10,20] the reactivity of the soluble I with $C_6H_5C{\equiv}CC_6H_5$ is not too surprising. However, it is remarkable that this reaction can be readily reversed: addition of THF to II generates a burgundy solution from which I and $C_6H_5C{\equiv}CC_6H_5$ can be recovered (Scheme I). This is the first evidence that the reversibility necessary for the development of catalytic cycles involving a Ln(II)/Ln(III) couple[4,10,22] is available with the very reactive Sm(II).

A black hexane solution of II reacts with H_2, turning orange within an hour and depositing an orange precipitate. Separation of the solid by filtration, extraction with toluene, and recrystallization provide red-orange prisms, III, which have an empirical formula $[C_5(CH_3)_5]_2SmH$ on the basis of analytical, magnetic, spectral, and chemical data.[23] We previously postulated the existence of such a complex in the reaction mixture obtained in the synthesis of I,[10] but we were unable to isolate it in that case. Indeed, the thermal stability of III is limited to 1–2 days in solution and 5–7 days in the solid state. Nevertheless, we were able to characterize III by X-ray diffraction (Figure 1).[24]

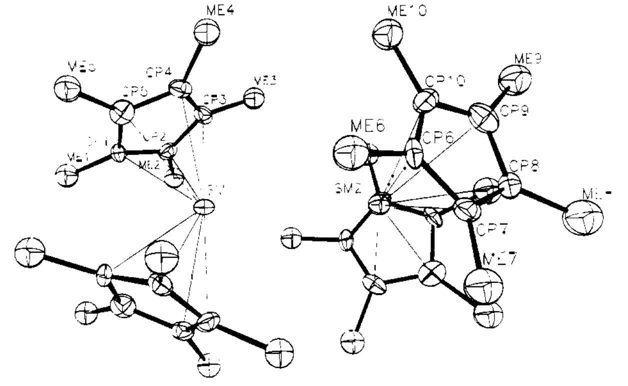


Figure 1. ORTEP plot of the molecular structure of $[(C_5Me_5)_2SmH]_2$.

III crystallizes as a dimer in which the two samarium atoms are on a crystallographic 2-fold axis and are separated by 3.905 (3) Å. This distance is too long for a Sm–Sm bond, but it is quite reasonable for a hydride-bridged structure on the basis of our recent characterization of $[(CH_3C_5H_4)_2Ln(THF)(\mu\text{-}H)]_2$, V (Ln = Y, Er).[11,25] A major difference between III and V is that the Ln_2H_2 plane in V bisects the Cp'(centroid)–Y–Cp'(centroid) angles for both Cp'_2Y units (Cp' = $CH_3C_5H_4$), whereas in III one $[C_5(CH_3)_5]_2Sm$ unit is rotated with respect to the other such that the molecule very nearly possesses an S_4 axis coincident with the crystallographic C_2 axis. The (ring 1 centroid)–Sm(1)–Sm(2)–(ring 2 centroid) dihedral angle is 87°. Hydrogen positions were not located due to the usual difficulty in finding hydrogen atoms near heavy metals.[27]

It is remarkable that III could be isolated and structurally characterized. Samarium complexes in general are more difficult to isolate than complexes of the smaller, later lanthanides such as Er and Lu, since it is more difficult to sterically saturate the coordination sphere of this larger metal to achieve stability.[28] Given the need for higher coordination numbers for the larger metal and considering the nine-coordinate THF-solvated Lu, Er, and Y species, complexes V,[11] and the nine-coordinate actinide hydrides $[(C_5Me_5)_2MH(\mu\text{-}H)]_2$ (M = Th, U),[29] in which the size of the cyclopentadienyl ligands is the same as in III (the Sm(III) radius is between the U(IV) and Th(IV) radii), one would expect at least a coordination number of nine for Sm in III.[30] A structure

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[15] In the heterogeneous reaction of lithium with $C_6H_5C{\equiv}CC_6H_5$, dimerization does occur: Braye, E. H.; Hubel, W.; Caplier, I. *J. Am. Chem. Soc.* **1961**, *83*, 4406–4413.

[16] Girard, P.; Namy, J. L.; Kagan, H. B. *J. Am. Chem. Soc.* **1980**, *102*, 2693–2703.

[17] Ananthanarayan, T. P.; Gallagher, T.; Magnus, P. *J. Chem. Soc., Chem. Commun.* **1982**, 709–710.

[18] $10^6\chi_g$ (cgs) = 2.38; μ_{eff}^{290} = 1.7 μ_B; near IR-vis (λ_{max}, nm (ϵ)) 1550 (22), 1535 (28), 1475 (30), 1440 (80), 1365 (62), 1230 (30), 1160 (18), 1150 (26), 1125 (28), 1010 (4); the remainder of the spectrum is dominated by a charge-transfer-like absorption.

[19] Compare the intense dark colors of the products of cocondensation of lanthanide atoms with dienes and alkynes.[5-8]

[20] Sm(III) + e → Sm(II), −1.55 V vs. NHE: Morss, L. R. *Chem. Rev.* **1976**, *76*, 827–841.

[21] A 30% yield of crystals of I is recovered by crystallization at −78 °C (cf. ref 10).

[22] Watson, P. L. *J. Chem. Soc., Chem. Commun.* **1980**, 652–653.

[23] Anal. Calcd for $SmC_{20}H_{31}$: Sm, 35.9. Found: Sm, 33.3. $10^6\chi_g$ (cgs) = 1.99; μ_{eff} = 1.4 μ_B; ^1H NMR (C_6D_6) δ 15.61 (br, SmH), −0.80 (s, $C_5(CH_3)_5$); IR (KBr) 1435 (s), 1375 (m), 1140 (s, v br), 1010 (m), 975 (m, br), 970 (w, br) cm^{-1}. III exchanges with D_2 to form an analogous deuteride which lacks the δ 15.61 resonance in the ^1H NMR spectrum and has a reduced $C_5(CH_3)_5$ signal due to extensive incorporation of deuterium into the pentamethylcyclopentadienyl ligands (confirmed by GC–MS). The IR spectrum of the deuteride lacks the 1140-cm^{-1} absorption and contains a new strong absorption at 820 cm^{-1} (ν_H/ν_D = 1.40). Hydrolysis of III forms H_2 in 74% yield.

[24] Single crystals of III were obtained by diffusion of pentane into a toluene solution at −5 °C. Space group $C2/c$; cell constants a = 16.532 (6) Å, b = 14.260 (4) Å, c = 16.948 (4) Å, β = 104.26 (4)° with four dimers per unit cell and D_c = 1.48g cm^{-3}. Least-squares refinement on the basis of 1004 observed reflections led to a final $R = \sum(|F_o| - |F_c|)/\sum|F_o| = 0.041$. The diffraction experiment was conducted as described in Holton et al.: Holton, J.; Lappert, M. F.; Ballard, D. G. H.; Pearce, R.; Atwood, J. L.; Hunter, W. E. *J. Chem. Soc., Dalton Trans.* **1979**, 45–53. An empirical absorption correction was done according to Churchill and Hollander: Churchill, M. R.; Hollander, F. J. *Inorg. Chem.* **1978**, *17*, 1957–1962. Anisotropic thermal parameters were those in SHELX, a system of computer programs for X-ray structure determinations by G. M. Sheldrick, 1976. The methyl groups had high thermal motion, but thermal parameters were acceptable and R was virtually the same for either isotropic or anisotropic refinement of the methyls.

[25] The atomic radius of samarium in rhombohedral α Sm is 1.802 Å: Moeller, T. In "Comprehensive Inorganic Chemistry"; Bailar, J. C., Jr., et al., Eds.; Pergamon Press: Oxford, 1973; Chapter 44. The Y···Y distance in complex V is 3.664 (1) Å[11], and the Y^{3+} radius is 0.084 Å smaller.[26]

[26] Cotton, F. A.; Wilkinson, G. "Advanced Inorganic Chemistry", 4th ed.; Wiley: New York, 1980.

[27] Sm–C bond lengths in III varied from 2.72 (2) to 2.81 (2) Å with an average of 2.76 (3) Å.

[28] Evans, W. J. In "The Chemistry of the Metal–Carbon Bond"; Hartley, F. R., Patai, S., Eds.; Wiley: New York, 1982; Chapter 12.

[29] Fagan, P. J.; Manriquez, J. M.; Maata, E. A.; Seyam, A. M.; Marks, T. J. *J. Am. Chem. Soc.* **1981**, *103*, 6650–6667. Broach, R. W.; Schultz, A. J.; Williams, J. M.; Brown, G. M.; Manriquez, J. M.; Fagan, R. J.; Marks, T. J. *Science (Washington, D.C.)* **1979**, *203*, 172–174.

[30] Raymond, K. N.; Eigenbrot, C. W., Jr. *Acc. Chem. Res.* **1980**, *13*, 276–283.

for III containing doubly bridging hydrogens would have a maximum coordination number of eight. The structure of III clearly demonstrates the variability in coordination number that can occur in organo-f-element complexes even when radial sizes and ligands are very similar.[31]

Like **I**, complexes II and III can also be used to initiate the catalytic hydrogenation of alkynes. Interestingly, the catalytic rates for I–III differ substantially, suggesting that several catalytic pathways are possible in this system. II displays the highest turnover rates, 1.5 min^{-1} $[\text{Sm}]^{-1}$ for $C_6H_5C{\equiv}CC_6H_5$ to $C_6H_5CH_2CH_2C_6H_5$ and 3.7 min^{-1} $[\text{Sm}]^{-1}$ for 3-hexyne to hexane, which are the fastest observed for a homogeneous f-element-based system.[6,32] Mechanistic and structural studies on this system are continuing.

Acknowledgment. We thank the National Science Foundation for support of this research and the Camille and Henry Dreyfus Foundation for a Teacher–Scholar Grant (to W.J.E.).

Supplementary Material Available: Tables of bond distances, angles, final fractional coordinates, and thermal parameters (3 pages). Ordering information is given on any current masthead page.

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(31) Consistent with this variability, the differences in M···M distances, 0.102 Å, and in M–C(η^5) distances, 0.075 Å, between III and the thorium complex are much greater than the difference in their ionic radii, 0.026.[26]

(32) Bowman, R. G.; Nakamura, R.; Burwell, R. L., Jr.; Marks, T. J. *J. Chem. Soc., Chem. Commun.* **1981**, 257–258.

Total Synthesis of the Hypocholesterolemic Agent (+)-Compactin

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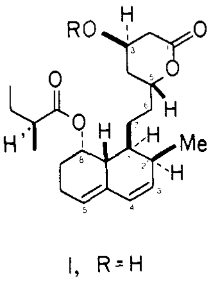
Received November 3, 1982

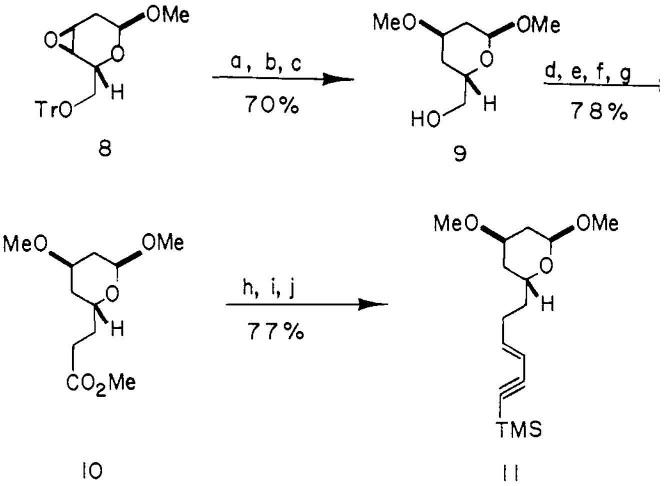
Compactin (**1**),[1] a fungal metabolite isolated from the strains of *Penicillium brevicompactum*, has been the object of intense synthetic activity[2,3] since the disclosure[4] that it is a potent competitive inhibitor of rat liver microsomal 3-hydroxy-3-methylglutaryl coenzyme A reductase, the rate-controlling enzyme in cholesterol biosynthesis. We detail in this communication a highly convergent, enantiospecific total synthesis of (+)-compactin.



I, R = H

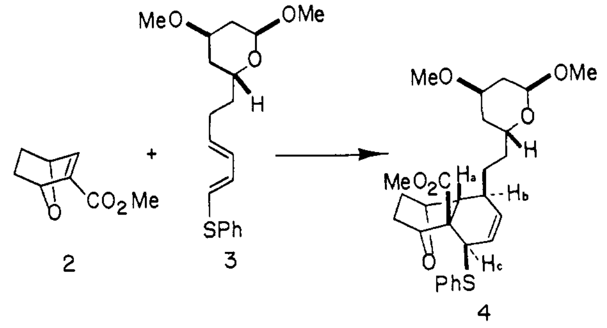
18, R = Me

Scheme I. Preparation of Enyne 11[a]

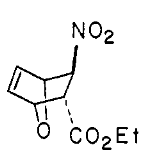


[a] (a) $LiAlH_4$, Et_2O, −10 °C, 3.5 h; (b) NaH, THF, MeI, 18 h; (c) Na, NH_3, −78 °C (30 min) → −33 °C (30 min); (d) TsCl, py, 11 h; (e) NaI, MEK, reflux, 4 h; (f) $C_6H_5SO_2CH_2CO_2Me$, Me_2SO Me_2SO^- Na^+, 80 °C, 9 h; (g) 6% Na(Hg), MeOH, Na_2HPO_4, 0 °C, 15 min; (h) $LiAlH_4$, Et_2O, 0 °C, 15 min; (i) $CrO_3{\cdot}2py$, CH_2Cl_2, 0 °C, 20 min; (j) $(C_6H_5)_3P^+CH_2C{\equiv}C(Me_3Si)\ Br^-$, BuLi, THF, −78 °C.

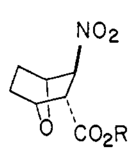
Our strategy for the construction of compactin centered around the Diels–Alder reaction between the chiral dienophile **2** and the chiral diene **3**, which provides in a single operation the intact carbon framework (cf. **4**) of **1**.



Synthesis of the 7-oxabicyclo[2.2.1]heptane derivative **2** in chiral form commenced with the known Diels–Alder adduct **5**,[5] readily available from furan and ethyl β-nitroacrylate.[6] Reduction ($K^{+-}OOCN{=}NCOO^-K^+$, MeOH, AcOH, 0 °C, 8 h) of the double bond in **5** followed by hydrolysis (10% KOH, THF, 0 °C) of the ester gave rise to a 92% yield of racemic acid **6**, mp 164–166 °C. Treatment of **6** with D(−)-α-phenylglycinol in acetonitrile containing 1.2 equiv of dicyclohexylcarbodiimide gave rise to a near quantitative yield of a diastereomeric mixture of sensitive amides, which were readily separated by HPLC.[7] The less polar



5



6 R = H

7 R = Me

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(1) Brown, A. G.; Smale, T. C.; King, T. J.; Hasenkamp, R.; Thompson, R. H. *J. Chem. Soc., Perkin Trans 1* **1976**, 1165. Also see: Endo, A.; Kuroda, M.; Tsujita, Y. *J. Antibiot.* **1976**, *29*, 1346. For a recent review of compactin's activity see: Endo, A. *Trends Biochem. Sci.* **1981**, *6*, 10.

(2) For syntheses of (+)-compactin, see: (a) Wang, N.-Y.; Hsu, C.-T.; Sih, C. J. *J. Am. Chem. Soc.* **1981**, *103*, 6538. (b) Hirama, M.; Uei, M. *Ibid.* **1982**, *104*, 4251.

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