

participate in hydrogen-bonded networks as neutral, mono-, and diprotonated species, but also because the conformational freedom allows optimum orientation in space of the functional groups. The acid–base reaction allows a conception for the preparation of a great variety of hydrogen-bonded organometallic crystalline compounds in which metal atoms in different oxidation and spin states can be combined within robust hydrogen-bonded superstructures. Furthermore, the abundance of polarized C–H groups on metal-coordinated ligands, such as C_5H_5 and C_6H_6 , make the use of C–H...O hydrogen bonds also profitable, in particular since they may be reinforced when hydrogen donors and acceptors belong to cations and anions, respectively.^[6]

Experimental Section

$FeACH_2$ (84 mg, 0.3 mmol) was dissolved in anhydrous THF (20 mL), and $(\eta^5-C_5H_5)_2Co$ (57 mg, 0.3 mmol) was added. $FeACH_2$ (71 mg, 0.26 mmol) was dissolved in anhydrous THF (16 mL), and $(\eta^6-C_6H_6)_2Cr$ (54 mg, 0.26 mmol) was added. The oxidation of cobaltocene and bis-benzene chromium was almost instantaneous. In both cases an orange precipitate was formed. After 1 h the slurries were filtered, and the orange powders were recrystallized from nitromethane to afford air-stable orange crystals of **1** and **2**.^[8] $(\eta^5-C_5H_5)_2Co$ (57 mg, 0.3 mmol) was dissolved in water (20 mL) and stirred until complete oxidation to yellow $[(\eta^5-C_5H_5)_2Co][OH]$ had occurred. $FeACH_2$ (8 mg, 0.03 mmol) was added to an aliquot (5 mL) of the solution and stirred for 15 min. The yellow-orange solution was filtered and evaporated to dryness, and the resulting solid was recrystallized from nitromethane to afford well-formed crystals of **3**.^[8]

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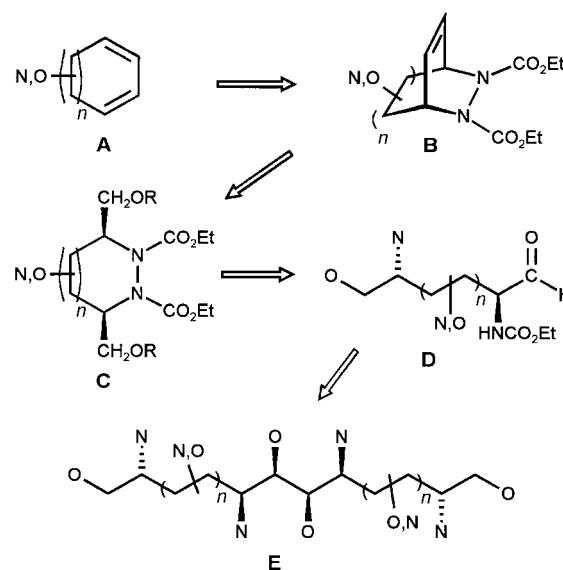
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Keywords: crystal engineering • hydrogen bonds • sandwich complexes • solid-state structures

From Cycloolefins to Linear C_2 -Symmetrical 1,4-Diamino-2,3-diol Building Blocks—Peptide Mimetics, Biocatalysis, and Pinacol Coupling of α -Amino Aldehydes**

Joachim Armbruster, Stefan Grabowski, Thomas Ruch, and Horst Prinzbach*

C_2 -Symmetrical 1,4-diamino-2,3-diol units of type **E** are central structural elements in numerous bioactive compounds.^[1] A variable synthesis of such building blocks, based on the recently reported route to enantiomerically pure amino aldehydes of type **D** from technical cycloolefins **A**,^[2] and



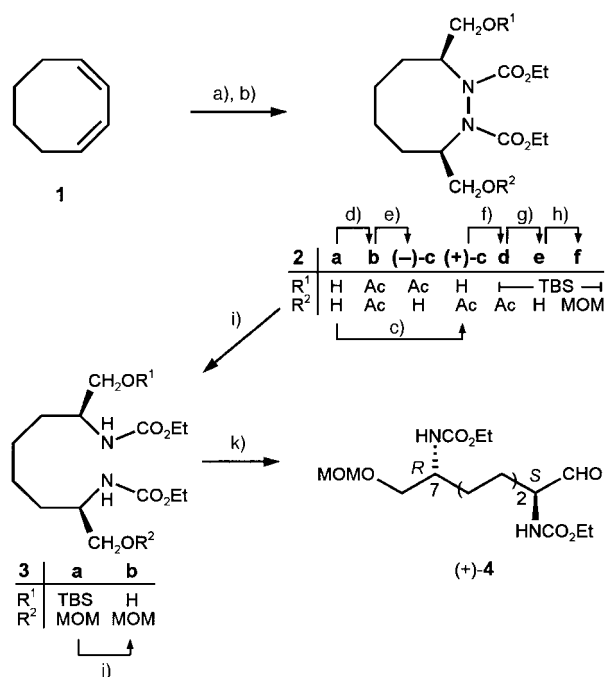
exemplary applications of this synthetic concept in the area of important bioactive compounds are the subjects of this communication.^[1, 3]

The linear, enantiomerically pure C_8 amino aldehyde (+)-**4** (**D**, $n=2$) is prepared by the reaction sequence depicted in Scheme 1:^[2] addition of diethyl azodicarboxylate (DEAD) to 1,3-cyclooctadiene (**1**), ozonolysis, reduction (strictly following a temperature program), and enzymatic asymmetrization, either by esterification of the *meso*-diethyl 3,8-bis(hydroxymethyl)-1,2-diazacyclooctane-1,2-dicarboxylate (**2a**) ($\rightarrow$ (+)-**2c**) or hydrolysis of the diacetate **2b** ($\rightarrow$ (-)-**2c**); lipozyme IM

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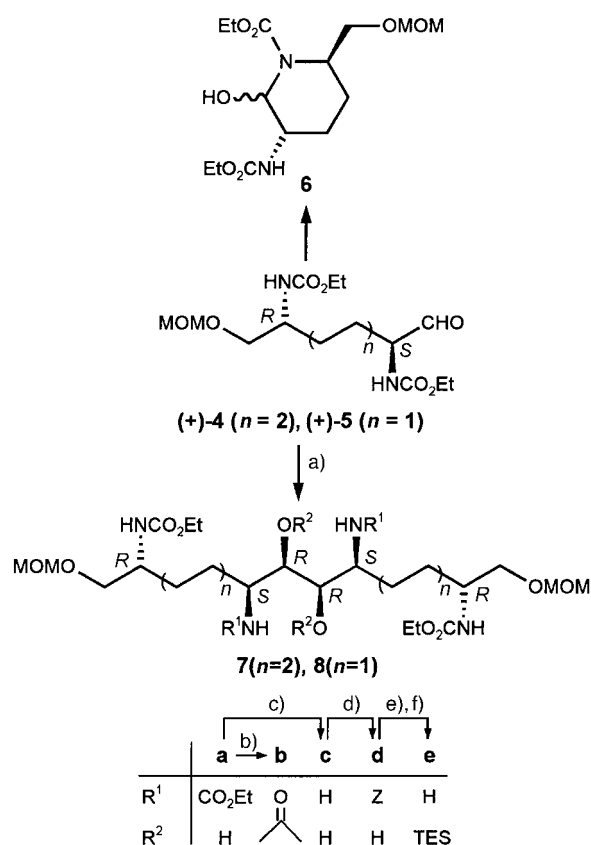
[**] Support by the Fonds der Chemischen Industrie and the BASF AG is gratefully acknowledged. J.A. and S.G. thank the Landesgraduiertenförderung Baden-Württemberg for a stipend. Thanks go to Dr. D. Hunkler for NMR, Dr. J. Wörth for MS, and G. Fehrenbach for HPLC analyses; Dr. H. Jendralla (Hoechst AG) for valuable advice; and Dr. L. Knothe for help with the manuscript.



Scheme 1. a) DEAD, *hv*, 5 d, 87%; b) O₃, EtOH, -48 °C, then NaBH₄, -48 °C, 10 min, room temperature (RT), quenching with acetic acid at -48 °C, 92%; c) lipzyme IM, vinyl acetate, RT, 4 d, 78%, *ee* > 95%; d) Ac₂O, pyridine, DMAP, RT, 12 h, 98%; e) lipzyme IM, phosphate buffer (0.2 N; pH 6.8), *n*-hexane, RT, 8 d, 69%, *ee* = 93%; f) TBSCl, imidazole, DMF, RT, 96%; g) NH₃/MeOH, 0 °C, 4 d, 98%; h) MOMCl, *i*Pr₂NEt, 0 °C, 1 d, 91%; i) Li/NH₃, -78 °C, 1 h, 93%; j) TBAF, THF, RT, 2 h, 98%; k) oxalyl chloride, DMSO, -78 °C, NEt₃, 1 h, quenching with citrate buffer (pH 3.3) at -15 °C, 96%. DEAD = diethyl azodicarboxylate, DMAP = 4-dimethylaminopyridine, TBS = *tert*-butyldimethylsilyl, MOM = methoxymethyl, TBAF = tetrabutylammonium fluoride, DMSO = dimethyl sulfoxide.

proved to be the enzyme of choice for both alternatives (70–80% yield each, *ee* ≥ 93%). Manipulation of the protecting groups (so far only practiced with (+)-2c to give 2f), reductive N–N cleavage (3a) as well as the final Swern oxidation 3b → (+)-4 (the absolute configuration 2*S*,7*R* is preliminary^[2]) proceeded with very high yield; under the specified conditions the optical purity is not affected.^[1c, 4] In crystalline form (+)-4 can be kept in stock, whereas in solution (CHCl₃) epimerization at C-2 occurs slowly.

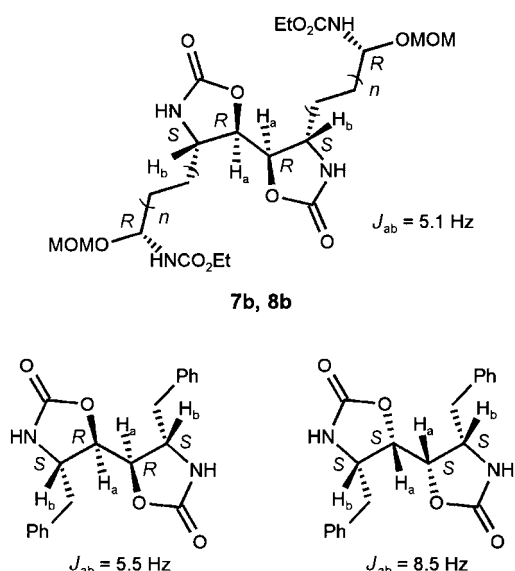
With aldehyde (+)-4 and the C₆ homologue (+)-5^[2] the prospect for stereoselective pinacol coupling was checked by making use of several one-electron reductants (e.g. SmI₂^[5] and C₆Mg^[6]).^[7] Caulton's reagent ([V₂Cl₃(thf)₆][Zn₂Cl₆])^[8] provided the best—for (+)-4 and (+)-5, however, significantly differing—results so far (Scheme 2): In the case of (+)-4 0.27 equiv of reagent (ca. 1 equiv of V²⁺) was sufficient to bring about total conversion. After appropriate workup (addition of the reaction mixture to 0.5 M citrate buffer, pH 4.7), the isolated mixture of dimers (>90%) consisted mainly of 7a; the yield of pure 7a (>80%) collected after crystallization from ethanol compares well with literature reports.^[1c, 4, 9] One of the two side products (ca. 5% each, “dimers” according to mass spectrometry) was identified as a O-MOM-cleaved derivative of 7a; for the second a reliable



Scheme 2. a) With (+)-4: 0.27 equiv of [V₂Cl₃(thf)₆][Zn₂Cl₆], CH₂Cl₂, degassed, anhydrous, N₂ atmosphere, RT, 2 h, then 0.5 M citrate buffer (pH 4.7), >90% mixture of diastereomers (>80% 7a); with (+)-5: a) 1.0 equiv of [V₂Cl₃(thf)₆][Zn₂Cl₆], CH₂Cl₂, RT, 4 h, then 0.5 M citrate buffer (pH 3.3), 60% mixture of diastereomers (80% 8a), 26% 6; b) 0.18 M NaOH in dioxane/water (1/1), RT, 2 h, 99%; c) 0.18 M NaOH in dioxane/water (1/1), 80 °C, 12 h; d) ZCl, *t*BuOH, NaHCO₃, H₂O, RT, 15 min, 78% (based on 7a); e) TESCl, imidazole, DMF, RT, 3 h; f) H₂, Pd/C (5%), toluene, 4 h, 92% (based on 7d). Z = benzyloxycarbonyl, TES = triethylsilyl.

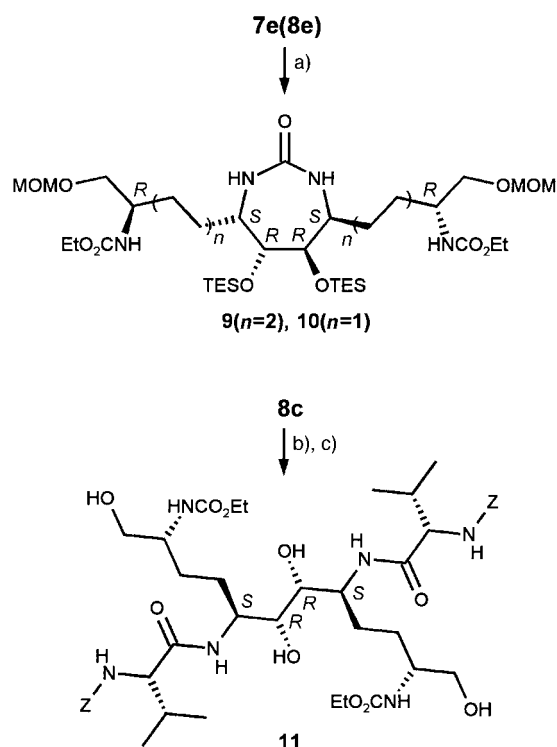
assignment is not yet possible (*S,S,S,S* dimer?). For total conversion of (+)-5 a significant excess of reagent is needed (1.0 equiv, that means 4 equiv of V²⁺). The outcome of the reaction is insofar much more complex, as besides dimerization to give 8a (ca. 45% in addition to ca. 10% of diastereomers) O-MOM cleavage (12%) and cyclization to the azasugar 6 (up to 26%) cannot be avoided (no hint of the *S,S,S,S* dimer). The amount of reagent necessary presumably reflects the complexations induced by the given functionalization pattern and responsible for the competing reaction channels.^[4, 8] The *S,R,R,S* configuration assigned to 7a and 8a is based on a comparison of the bisoxazolidinones 7b and 8b, synthesized from 7a and 8a under base catalysis, with the bisoxazolidinones pictured in Scheme 3, whose absolute configurations of *S,R,R,S* and *S,S,S,S* have been established,^[10] and on the evaluation of the diastereomeric transition states of the dimerization pathways (Pederson^[8]).

From 7a and 8a—examples for potential libraries of structurally varied derivatives—the cyclic ureas 9 and 10 as well as the pseudotetrapeptide 11 are prepared (Scheme 4); they are potential peptide mimetics (see, for example, the structurally related protease inhibitor models DMP 323^[1a] and



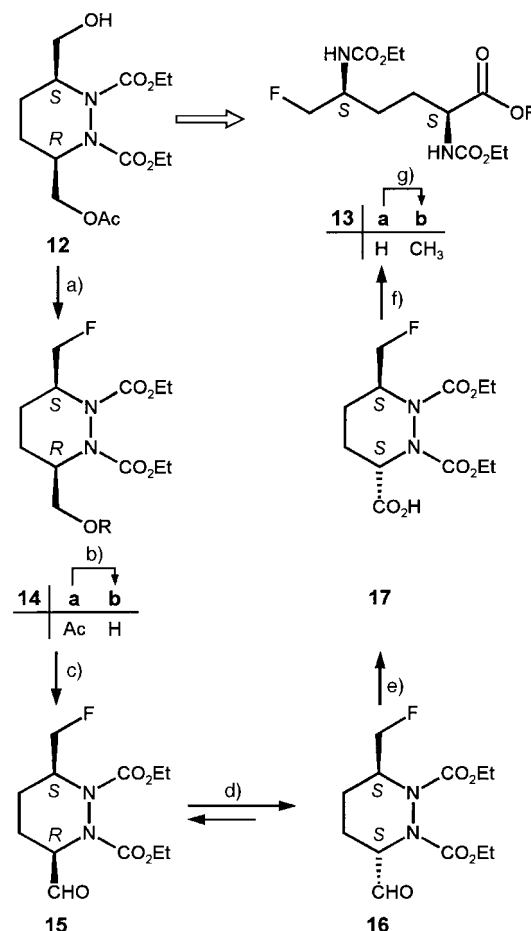
Scheme 3. The assignment of the absolute configuration of **7b** and **8b**.

A 77003^[1b]). It was essential for the efficiency of these syntheses that the central urethane units in **7a** and **8a** could be selectively saponified to **7c** and **8c** with anchimeric assistance of the OH groups. The subsequent steps (**c–e**) to yield **9** and **10** were carried out following known procedures and were unproblematic;^[1c] for the condensation of the highly polar, even in DMF hardly soluble **8c** with *Z*-L-valine, a protocol guaranteeing a low degree of racemization was helpful [activation with the uronium salt TOTU ((Me₂N)₂CON=C(CN)CO₂Et)⁺ BF₄[–])].^[11]



Scheme 4. a) CDI, toluene, RT, 8 h, 53–58%; b) *Z*-valine, *i*Pr₂NEt, TOTU, DMF, RT, 1 h, 67% (based on **8c**); c) MeOH, HCl, 60°C, 2 h, quant. CDI = 1,1'-carbonyldiimidazole.

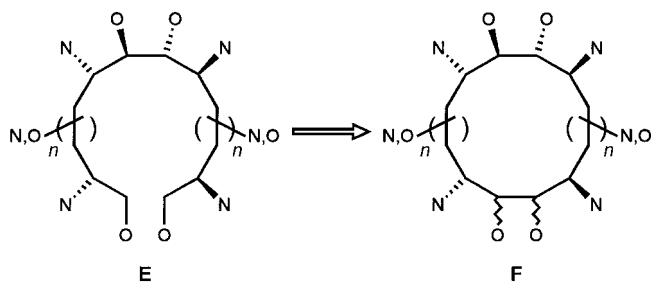
The stereochemical variability of the synthetic concept **A** → **B** → **C** → **D** linked with the 1,2-diazacyclic intermediates of type **C** (*n* = 1)^[2] is exemplified by the advantageous route from **12** to (2*S*,5*S*)-5-fluoromethylornithine (**13a**, an inhibitor of ornithine aminotransferase^[12]) depicted in Scheme 5. Under stereochemical aspects, it is decisive that in the base-



Scheme 5. a) DAST, CH₂Cl₂, RT, 30 min, 74% **14a**; b) MeOH/NH₃ (aq) (4/1), 0°C, 24 h, 80% **14b**; c) oxalyl chloride, DMSO, NEt₃, CH₂Cl₂, –78°C, 45 min, quant.; d) *i*Pr₂NEt, dioxane, 70°C, 12 h; e) NaClO₂, 2-methyl-2-butene, *t*BuOH, aq phosphate buffer, RT, 16 h, 89%; f) Li, NH₃ (aq), THF, –78°C, 15 min, **13a**; g) CH₂N₂, dioxane, RT, 15 min, ca. 50% **13b** (based on **17**). DAST = (diethylamino)sulfur trifluoride.

catalyzed *cis/trans* equilibrium between the 3,6-disubstituted hexahydropyridazine diesters *cis*-**15** (3*a*6*e* ⇌ 3*e*6*a*) and *trans*-**16** (3*e*6*e* ⇌ 3*a*6*a*) the latter is highly favored owing to the thermodynamic advantage of axial substitution (NMR;^[13] in support of this, lactonization is fast in *cis*-**14b**, but not in the *trans*-epimer generated from **16**). Whereas the oxidation **16** → **17** with NaClO₂ is straightforward, optimization is still necessary for the subsequent steps **17** → **13b** (preliminarily ca. 50%). This synthetic strategy is clearly applicable to analogous 2*R*,5*R* and 2*R*,5*S* structures.

Further functionalization of the (CH₂CH₂)_{*n*} elements implied in formula **A**, as well as cyclization of the dimers (e.g. **E**) available through this synthetic strategy to the



poly(per)functionalized macrocycles (e.g. **F**), are intensively pursued projects.

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Keywords: amino aldehydes • dimerizations • enzyme catalysis • enzyme inhibitors • peptidomimetics

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The π -Pyrrole Complexation of Alkali Metal Ions by Zirconium *meso*-Octaalkylporphyrinogens: Encapsulation of Li₄H₄ and Li₂O in Sandwich Structures**

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Much attention has been focused on methodologies that allow ionic compounds to be carried into a nonpolar organic phase, either as an ion pair or in an ion-separated form.^[1] For alkali metal salts and polar organometallic compounds, two

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