2.50 (dd, $J = 1.6$, 16.2 Hz, 1H), 1.89–1.93 (m, 1H), 1.73–1.81 (m, 2H), 1.60–1.63 (m, 1H), 1.55 (brs, 1H, OH), 1.28–1.40 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): $\delta = 143.47$, 129.73, 126.68, 119.18, 116.49, 113.43, 72.67, 53.93, 33.16, 32.96, 27.80, 25.31, 23.35; HR-MS (CI): calcd (found) for $C_{13}H_{17}NO$ [M^+]: 204.1388 (204.1397).

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[7] a) Crystal structure data for **4**: $C_{12}H_{13}NO_2$; monoclinic; $P2_1c$; $a = 14.972(7)$, $b = 5.599(3)$, $c = 13.403(5)$ Å; $\alpha = 90$, $\beta = 114.93(3)$, $\gamma = 90°$; $Z = 4$; R_1 [$I > 2\sigma(I)$] = 0.0386; b) crystal structure data for **8**: $C_{14}H_{15}NO_2$; monoclinic; $P2_1c$; $a = 17.7653(11)$, $b = 9.1414(6)$, $c = 15.4356(10)$ Å; $\alpha = 90$, $\beta = 115.5670(10)$, $\gamma = 90°$; $Z = 8$; R_1 [$I > 2\sigma(I)$] = 0.0481; c) crystal structure data for **6**: $C_{12}H_{12}NO_2Cl$; monoclinic; $P2_1n$; $a = 6.2128(5)$, $b = 12.1974(12)$, $c = 14.4788(13)$ Å; $\alpha = 90$, $\beta = 98.188(3)$, $\gamma = 90°$; $Z = 4$; R_1 [$I > 2\sigma(I)$] = 0.0601. Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication nos. CCDC-113239/113240/113241. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).
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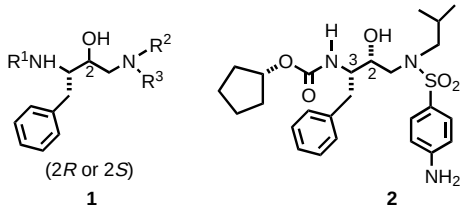
[11] The substrates **3**, **5**, and **7** were prepared starting from the corresponding allylic alcohols by the sequence: 1) conversion into chloroformates by reaction with phosgene, 2) treatment of the chloroformates with *o*-aminobenzyl alcohol, and 3) conversion of the resulting hydroxy carbamates into the corresponding chlorides with thionyl chloride (see experimental section for details). 3-Chloro-2-methyl-2-propen-1-ol was prepared according to A. Mooradian, J. B. Cloke, *J. Am. Chem. Soc.* **1946**, *68*, 785–789; L. F. Hatch, J. J. Russ, L. B. Gordon, *J. Am. Chem. Soc.* **1947**, *69*, 2614–2616. Enantiomerically pure (*R*)-2-cyclohexen-1-ol was prepared by kinetic resolution with lipase as described by T. Fukazawa, T. Hashimoto, *Tetrahedron: Asymmetry* **1993**, *4*, 2323–2326.

re- and *si*-Face-Selective Nitroaldol Reactions Catalyzed by a Rigid Chiral Quaternary Ammonium Salt: A Highly Stereoselective Synthesis of the HIV Protease Inhibitor Amprenavir (Vertex 478)**

E. J. Corey* and Fu-Yao Zhang

The development of therapeutically useful HIV protease inhibitors has been one of the major contributions of synthetic and medicinal chemistry to human well-being during this decade.[1, 2] Since several of these agents possess chiral substituted 1,3-diamino-2-hydroxypropyl segments as a central structural subunit, for example, the phenylalanine-related subunit **1**, considerable research effort has been directed at methods for their synthesis.[3, 4] Described herein is a new strategy for stereocontrol in the synthesis of subunits of type **1** and a specific application with a practical synthesis of amprenavir (**2**),[5, 6] an important second generation HIV



protease inhibitor with a number of clinical advantages over first generation agents. The method currently being used to produce **2** involves the activation of the carboxyl group of *N*-*tert*-butoxycarbonylphenylalanine and transformation to the corresponding diazomethyl ketone with diazomethane as a key element.[7] Because of the intrinsic hazards of this step a safer alternative seems preferable.

The sequence utilized for the synthesis of amprenavir is summarized in Scheme 1. *N*,*N*-Dibenzyl-(*S*)-phenylalaninal (**3**)[8] in tetrahydrofuran (THF) was added to a mixture of quaternary ammonium salt **4**,[9, 10] nitromethane, and finely divided potassium fluoride in THF with stirring. After 6 h the nitro alcohol **5** was isolated in 86% yield by flash chromatography on silica gel.[10] The more polar C(2) diastereomer of **5** was isolated in 5% yield, which indicated a 17:1 diastereoselectivity for the nitroaldol reaction.[11] In contrast, the nitroaldol reaction of **3** in the presence of tetra-*n*-butylammonium fluoride[8c] afforded a mixture of **5** and the C(2) diastereomer with only 4:1 diastereoselectivity under the same reaction conditions. Treatment of nitro alcohol **5** with 2.5 equivalents of $NiCl_2$ and 25 equivalents of $NaBH_4$ in methanol at 0 °C for 10 min with vigorous agitation provided

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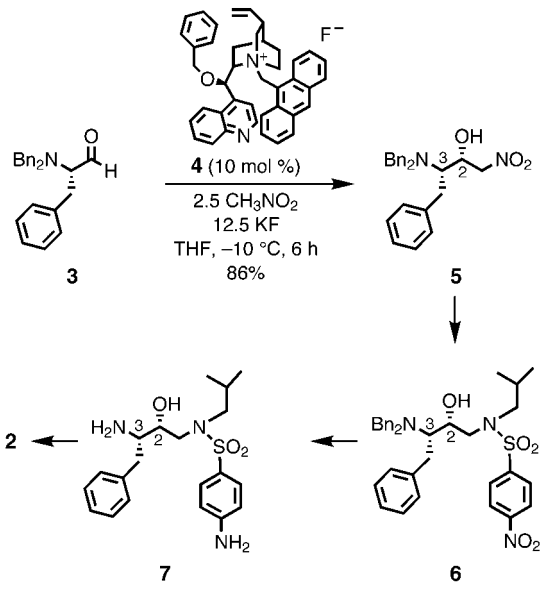
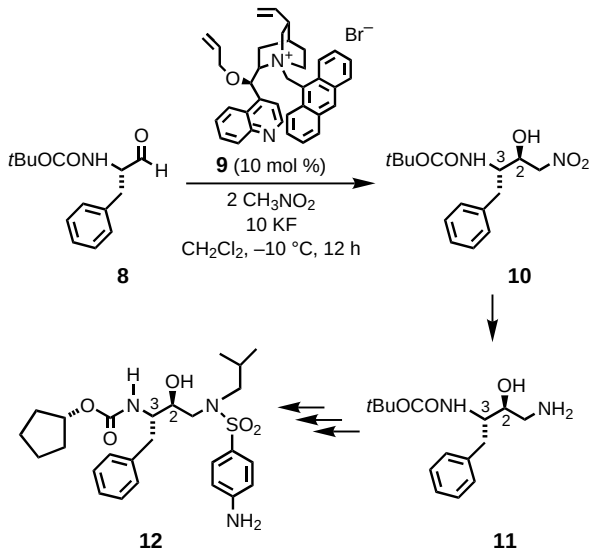


Scheme 1. Application of the stereoselective nitroaldol reaction promoted by a chiral quaternary ammonium salt to the synthesis of amprenavir (**2**).



Scheme 2. Application of the stereoselective nitroaldol reaction promoted by chiral quaternary ammonium salts to the synthesis of the C(2) diastereomer **12** of amprenavir.

the corresponding amino alcohol (85%), which was reductively alkylated by reaction with isobutyraldehyde/$MgSO_4$ to form the Schiff base, which was subsequently exposed to sodium borohydride in ethanol at 0–23 °C for 4 h. Reaction of the resulting secondary amine (82% yield)[12] with *p*-nitrobenzenesulfonyl chloride/Et_3N in CH_2Cl_2 at 23 °C for 2 h produced sulfonamide **6** (94%). Amprenavir (**2**) was synthesized from **6** in 95% overall yield by: 1) catalytic hydrogenation (1 atm H_2, $Pd(OH)_2$/C, MeOH, 23 °C for 4 h) to form diamine **7** and 2) reaction of **7** with (*S*)-3-tetrahydrofuranyl-*N*-oxysuccinimidyl carbonate and Et_3N in CH_2Cl_2 at 23 °C for 1 h. The overall yield of **2** from amino aldehyde **3** was 50%. Comparison of the spectroscopic data of the product with those for authentic amprenavir confirmed the identity of our synthetic **2**.

The nitroaldol reaction promoted by chiral quaternary ammonium fluoride was also applied to the stereoselective synthesis of the 2*S*,3*S*-diastereomeric series starting from the *N*-*tert*-butoxycarbonyl derivative of *S*-phenylalaninal (**8**, Scheme 2).[9b] A mixture of **8**, 10 mol% of quaternary ammonium salt **9**,[13] and 10 equivalents of finely powdered KF in CH_2Cl_2 was treated with 2 equivalents of nitromethane at –10 °C with stirring. After 12 h the organic product, which consisted of a 9:1 mixture of the 2*S*,3*S* nitro alcohol **10** and the 2*R*,3*S* diastereomer 2-*epi*-**10** (88% total yield), was purified by flash chromatography on silica gel to give pure samples of **10** and 2-*epi*-**10**. Reduction of **10** (1 atm H_2, Pd/C, EtOH, 23 °C, 5 h) produced amino alcohol **11** (87%) and similarly reduction of 2-*epi*-**10** afforded 2-*epi*-**11**. The latter was identified by comparison with an authentic sample.[7b, 14] Amino alcohol **11** was transformed to the C(2) diastereomer of amprenavir (**12**) by using the methods described above for the synthesis of amprenavir. In contrast to the stereoselectivity observed for the conversion **8** → **10** under the influence of the chiral salt **9**, the reaction of **8** and nitromethane in the presence of *n*-Bu_4NF or achiral amines afforded approximately 1:1 mixtures of **10** and the C(2) diastereomer.

The synthetic utility of the chiral quaternary ammonium salts for enantioselective alkylation and Michael addition reactions, as described previously, depends on the three-dimensional rigidity of the cation in solution and the preference for one particular conformation.[9] This conformation suggests a working mechanistic model that provides a plausible explanation for the novel face-selective nitroaldol reactions reported herein. In the case of the *re*-face-selective nitroaldol reaction **3** → **5**, there is one particular assembly of the aldehyde **3** with cation **4** that places the formyl oxygen atom in proximity to the sole accessible face[9] of the ammonium nitrogen atom (leading to a contact ion pair, $-O^{\delta-}\cdots N^+$, in the transition state for addition of $CH_2NO_2^-$ to the formyl group), while affording maximum van der Waals contact between **3** and **4**. A graphical rendering of the three-dimensional structure of this assembly of **3** and **4** is shown in Figure 1. The carbon atoms of the aldehyde **3** are darkened relative to the carbon atoms of the cation **4**. In the front view the *re* face of the formyl group is exposed, the *si* face is behind, and the formyl oxygen atom sits right above the ammonium N^+ ion (not visible). One of the benzyl groups of **3** makes a face–face contact with the anthracene subunit of **4** (see back view) and the other contacts the quinoline ring of **4**. As a result of the snug fit between **3** and **4** in the assembly shown in Figure 1 only the *re* face of **3** is exposed to attack by $CH_2NO_2^-$ and the proximity of the N^+ ion to the formyl oxygen atom of **3** promotes the carbonyl addition through a charge acceleration.

Figure 2 depicts an assembly of the aldehyde **8** and ammonium cation **9** that allows van der Waals contact between the *tert*-butyl and phenyl groups of **8** and the 9-anthracenylmethyl subunit of **9**, while placing the formyl oxygen atom of **8** in proximity to the open face of the N^+ center of **9**. The attack of $CH_2NO_2^-$ on the formyl carbon atom in this assembly must occur at the *si* face of the CHO group, a reaction pathway that leads to the predominating nitroaldol product **10**.

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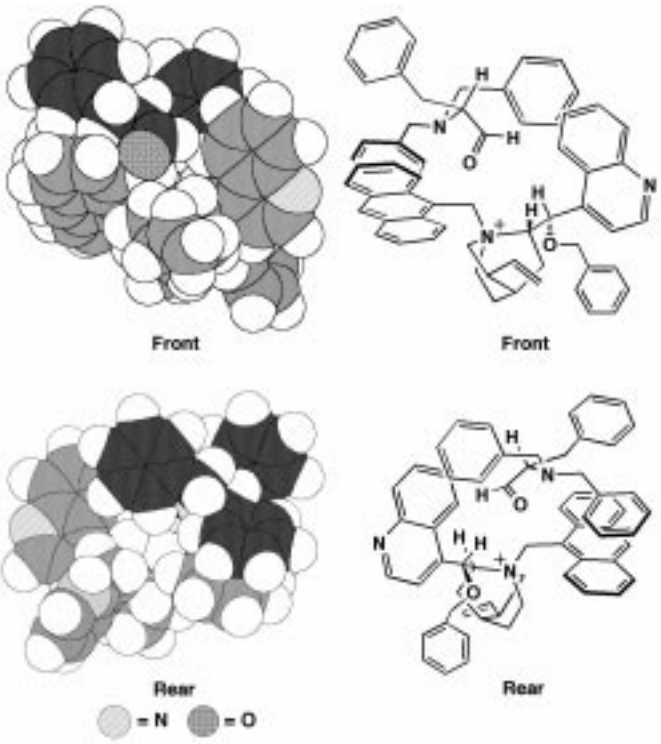

Figure 1. Two views of the assembly of **3** and **4** that could lead to nitroaldol product **5**. The front and rear views are related by a rotation of 180° about the vertical axis.

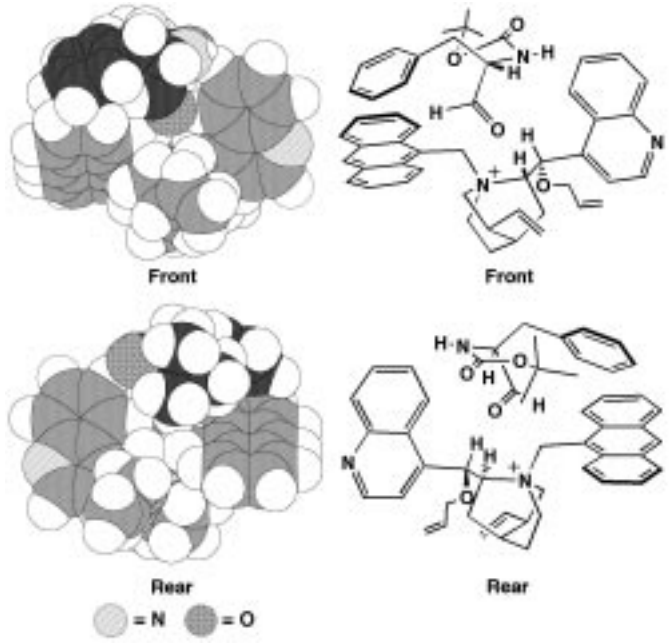

Figure 2. Two views of the assembly of **8** and **9** that could lead to nitroaldol product **10**. The two views are related by 180° rotation about the vertical axis.

Alternative pathways involving the reaction of a contact ion pair[9] of $CH_2NO_2^-$ and the chiral quaternary ammonium ion with external aldehyde cannot be excluded. However, such a mechanism does not lead to a clear explanation of the high stereoselectivities observed for the nitroaldol reactions **3** → **5** and **8** → **10**.

The use of the rigid chiral quaternary ammonium cations **4** and **9** to control the face selectivity in nucleophilic addition to aldehydes **3** and **8**, *re* or *si* depending on the N-protecting group of the *S*-phenylalaninal moiety, suggests a new strategy for stereocontrol of such reactions. Although further research is required to establish the scope and to validate the stereochemical/mechanistic model, the results reported herein and previously[9] (and the experimental simplicity) provide ample incentive for additional study. The experimental procedure for these stereoselective nitroaldol reactions is exceedingly simple (see Experimental Section).

Experimental Section

5: A well-stirred mixture of **4** (12 mg, 0.02 mmol), potassium fluoride (145 mg, 2.5 mmol), tetrahydrofuran (0.3 mL), and nitromethane (27 μL, 0.5 mmol) was cooled to −10 °C and treated with a solution of (*S*)-*N*,*N*-dibenzylphenylalaninal (66 mg, 0.2 mmol) in THF (0.4 mL). After stirring the mixture at −10 °C for 6 h the mixture was filtered to remove KF and concentrated. The solid quaternary ammonium salt **4** was precipitated with diethyl ether/hexane (7/3) and the product-containing soluble fraction was evaporated in vacuo to give an oil, which was purified by flash chromatography (silica gel, hexanes/ethyl acetate (10/1)) to give the 2*R*,3*S* nitro alcohol **5** (66 mg) and the 2*S* diastereomer (4 mg). **5**: $[\alpha]_D^{23} = +12.5$ ($c = 1.1$ in $CHCl_3$); IR (film): $\tilde{\nu} = 3565.7, 3490.4, 3085.7, 2929.1, 2843.0, 2808.6, 1602.5, 1552.3, 1494.7, 1454.1, 1381.8, 1264.5, 1181.9\ cm^{-1}$; ^1H NMR (400 MHz, $CDCl_3$, 25 °C): $\delta = 7.29-7.10$ (m, 15H), 4.72 (dd, $J = 13.4, 2.0$ Hz, 1H), 4.42 (m, 1H), 3.93 (dd, $J = 9.7, 13.4$ Hz, 1H), 3.72 (d, $J = 13.5$ Hz, 2H), 3.44 (d, $J = 13.5$ Hz, 2H), 3.09 (m, 1H), 2.96 (dd, $J = 6.2, 10.5$ Hz, 2H), 2.29 (d, $J = 5.5$ Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): $\delta = 140.2, 138.8, 129.3, 128.8, 128.5, 128.4, 127.2, 126.2, 79.9, 70.6, 61.6, 54.6, 32.3$. HRMS (CI): calcd for $C_{24}H_{28}N_2O_3$ $[M+H^+]$: 391.2022, found: 391.2030.

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Keywords: amino alcohols · chiral auxillaries · diastereoselectivity · nitroaldol reaction · phase-transfer catalysis

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ethanol/water (1/2) at 65 °C (92 %) followed by oxidation with pyridine/SO_3 complex and Et_3N in dimethyl sulfoxide at 10 °C (97 %).
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[10] Quaternary ammonium fluoride **4** was prepared from the corresponding bromide[9] by passage of a methanolic solution through a column of ion-exchange resin Amberlyst A-26 (OH^-) to afford the corresponding quaternary ammonium hydroxide followed by treatment with one equivalent of HF and removal of solvent in vacuo. The quaternary ammonium salt may be separated from the nitro alcohol **5** for reuse by selectively dissolving the latter in diethyl ether.
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[13] The allyl ether quaternary ammonium salt **9** afforded somewhat better stereoselectivity in this reaction than did the benzyl ether analogue **4**. Bromide **9** is converted into the corresponding fluoride in situ.
[14] We are grateful to Dr. Peter Cheng of the Bristol – Myers Squibb Research Institute (Princeton, NJ) for an authentic sample of 2-*epi*-**11**[7b] and spectral data.

A Three-Dimensional Coordination Polymer with an Expanded NbO Structure**

Tianyan Niu, Xiqu Wang, and Allan J. Jacobson*

Coordination chemistry allows a systematic approach to the synthesis of extended lattices.[1] Many structures have been reported with unprecedented lattice types, while others are based on frameworks that are found in simple inorganic structures, for example, diamond,[2] PtS,[3] and quartz.[4] Several coordination polymers have been described[5] containing square-planar centers based on the NbO net (6^48^2), the tetragonal $CdSO_4$ structure (6^58, B), and the "dense" net (7^59, C). An example of an NbO net[6] (Figure 1) is the compound formed by cyanuril acid and biuret containing two interpenetrated hydrogen-bonded networks.[7,8] Examples of the other two structure types are the interpenetrated networks formed by copper(II) nitrate with 1,2-bis(4-pyridyl)ethane (B)[9] and 1,2-bis(4-pyridyl)ethyne (C).[5a] Schindler and Baur[10] have shown that several non-interpenetrated inorganic frameworks can be related to the NbO-type structure.

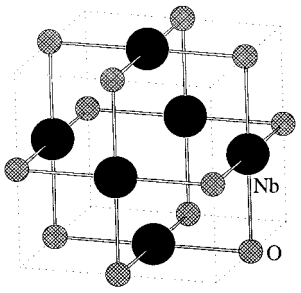

Figure 1. The framework of NbO.

For example sodalite, $[(Me_4N)_{1.3}(H_3O)_{0.7}\{Mo_4O_8(PO_4)_2\}]\cdot 2H_2O$,[11] and $[Cs_3\{V_5O_9(PO_4)_2\}]\cdot x\,H_2O$[12] frameworks can be constructed by connecting four rings of silicate tetrahedra $Si_4O_4O_{8/2}$, $Mo_4O_8(PO_4)_{4/2}$ cubes, and $V_5O_9(PO_4)_{4/2}$ "helmets", respectively, with 90° rotations between adjacent groups imposed by the shared tetrahedral bridging units.

We and others have investigated the syntheses and properties of coordination polymers formed by linking cyanometalate anions with trialkyl- or triaryltin cations.[13–18] The compounds in this class have the general formula $[(R_3Sn^{IV})_nM(CN)_m]$ and contain polymeric −M−C≡N−Sn−N−C≡M− chains which are connected to give frameworks of various topologies. A summary of the transition metals and organic R groups that have been used in the previous studies is given elsewhere.[16] This class of compounds has the potential for use in molecular separations[19] because large cavities are formed in some examples. Intercalation of large molecules, for example, ferrocene in $[(Me_3Sn)_3Fe(CN)_6]$[20] has been reported.

In compounds formed by square-planar $[Ni(CN)_4]^{2-}$ anions and R_3Sn^+ cations in a 1:2 ratio, two possible structure types can be anticipated. The first is a two-dimensional layer structure in which all $Ni(CN)_4$ planes are parallel. The second is a hypothetical framework that can be thought of as being related to the NbO structures discussed above, but expanded by the bridging R_3Sn^+ cations. This would entail alternating parallel and perpendicular $Ni(CN)_4$ planes. Here we report the synthesis of **1**, to our knowledge the first three-dimensional cyanometalate coordination polymer with this expanded NbO-type structure. The framework of **1** is not interpenetrated, and the large central cavity in the structure is filled by inclusion of Ph_3SnOH and solvent molecules during synthesis.

$$[(Ph_3Sn)_2Ni(CN)_4\cdot Ph_3SnOH\cdot\approx 0.8\,MeCN\cdot\approx 0.2\,H_2O] \qquad \mathbf{1}$$

Compound **1** was prepared in single-crystal form by slow interdiffusion of solutions of Ph_3SnCl in acetonitrile and $K_2[Ni(CN)_4]$ in water. The structure of **1** was determined by single-crystal X-ray diffraction,[21] and the composition was determined by elemental analysis. The local coordination environments of Ni and Sn atoms are shown in Figure 2. The

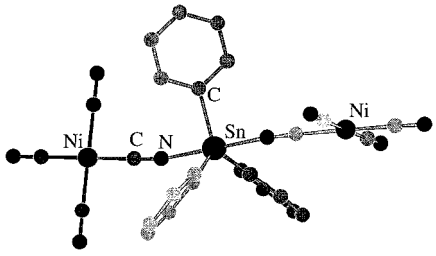

Figure 2. The local coordination environments of the nickel and tin atoms in **1**.

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