Complexes of P,N Macrocycles

A Strategy for the Synthesis of Phosphorus-Containing Macrocycles—Ligands for Exceptional Coordination Geometries**

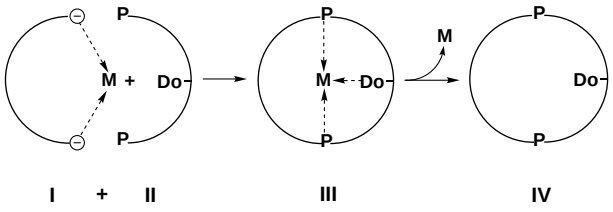
*Sahir Ekici, Martin Nieger, Robert Glaum, and Edgar Niecke**

In memory of Othmar Stelzer

On the basis of its simple implementation and high efficiency template-directed synthesis is of central importance for the assembly of macrocyclic structures.[1] Milestones of this discipline, which is situated on the interface between organic and supramolecular chemistry, include the syntheses of crown ethers, cryptands, and polyazamacrocycles which have potential applications in catalysis and electron transfer.[2] The macrocyclization is realized by a condensation or oxidative coupling reaction, where in particular the ions of alkali metals and late transition metals, respectively, proved their general usability.[1]

Template-directed syntheses of phosphamacrocycles normally proceed by 1,2-addition of primary or secondary phosphanes to vinylic or allylic double bonds, respectively.[3] However, examples of ring-closure reactions by alkylation of *cis* oriented PH units bound to a transition metal[4] or by reactions of bipodal, aliphatic, secondary phosphanes with β-carbonyl compounds are also present in the literature.[5] Unfortunately the access to such compounds is severely restricted by the limited availability of PH functionalized phosphanes.[6] Macrocycles containing additional donor atoms (N, O, S) besides the phosphorus fragment were obtained only in particular cases using high-dilution conditions or condensation reactions; template-directed ring closure is the exception.[7]

In the context of the synthesis of tridental hybrid ligands containing a PC_2EC_2P (E = N, O, S)[8,9] backbone with low-coordinated phosphorus centers we succeeded in developing a novel synthetic approach to macrocyclic structures. The strategy is based on a coupling reaction between two alkali-metal-fixed, carbanionic centers **I** (template) and two specifically connected, electrophilic phosphorus centers **II** (substrate). Twofold P–C bond formation first results in a metallamacrocycle **III** (in the form of a phosphamethanide complex), that is subsequently converted by protonation into the neutral ring system **IV** (Scheme 1). The reaction sequence



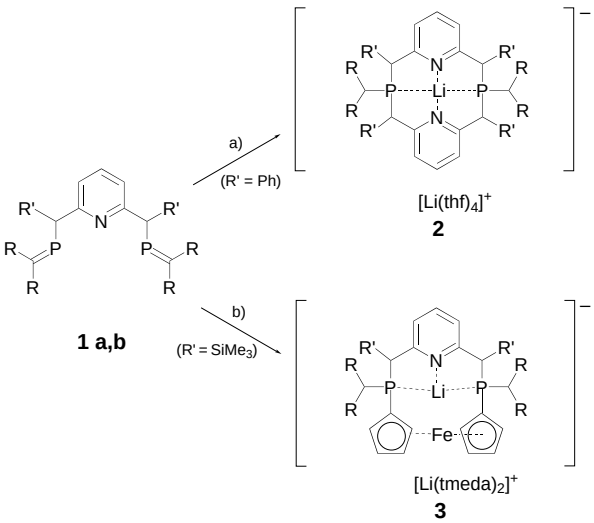
Scheme 1. Concept for the synthesis of macrocycles with phosphorus donor centers.

I + II→III→IV is supported by two examples: the synthesis of a) a 2,11-diphospha[3.3]-2,6-pyridinophane and b) a 1,10-diphospha(2)-2,6-pyridino(2)-1,1′-ferrocenophane incorporating a redox active metal center (Fe^{II}/Fe^{III}) in the framework.

The equimolar reaction of the bis(methylene)phosphane compounds **1a**[10] (R = $SiMe_3$, R′ = Ph) or **1b**[9](R = R′ = $SiMe_3$) with the lithium salts obtained from the deprotonation of 2,6-dibenzylpyridine and ferrocene, respectively,[11] yielded the respective ate complexes that were isolated in the form of their respective lithium salts **2** and **3** as red crystals (Scheme 2). Protonation (Et_3NHCl/THF) transforms the ate-complexes **2** and **3** to the respective neutral macrocycles **4** and **5**, that were also isolated as crystalline solids (Scheme 3).[12] The diazapyridinophane analogues of **4** and **5** are well documented in the literature.[13]

Starting from **4** a mononuclear, cationic complex can be obtained by reaction with $(Cu^{I}OTf)_2 \cdot C_6H_6$ (OTf = $CF_3SO_3^-$), which could be isolated as the yellow, crystalline salt **6**-OTf. Compound **5** reacts with tetracarbonyl nickel under displacement of three molecules of CO to yield the complex **7** as a red, crystalline solid (Scheme 3).

Figure 1 depicts the molecular structure of the anion **3**.[14,15] The macrocycle adopts a *syn*-conformation with a boat/



Scheme 2. Synthesis of **2** (**1a, 2**: R = $SiMe_3$, R′ = Ph) and **3** (**1b, 3**: R′ = R = $SiMe_3$); a) 2,6-di(lithiumbenzyl)pyridine, THF −78 °C; b) 1,1′-dilithiumferrocene, Et_2O/tmeda, −78 °C. tmeda = *N,N,N′*,N′-tetramethyl-1,2-ethanediamine.

[*] Prof. Dr. E. Niecke, Dipl.-Chem. S. Ekici, Dr. M. Nieger, Prof. Dr. R. Glaum
Anorganisch Chemisches Institut
Universität Bonn
Gerhard-Domagk-Strasse 1, 53121 Bonn (Germany)
Fax: (+49) 228-73-5792
E-mail: e.niecke@uni-bonn.de

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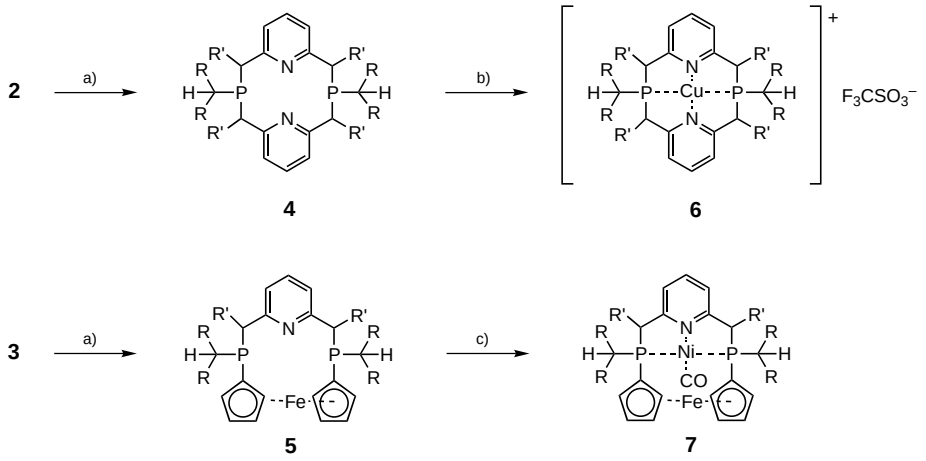

Scheme 3. Syntheses and reactions starting from **4** (**2, 4, 6**: R = $SiMe_3$, R′ = Ph) and **5** (**3, 5, 7**: R,R′ = $SiMe_3$); reagents: a) Et_3NHCl (2 equiv), THF, 0 °C; b) $(CuOTf)_2C_6H_6$ (0.5 equiv), toluene, RT; c) $[Ni(CO)_4]$, Et_2O, −30 °C.

boat-arrangement of the phosphorus atoms. The lithium atom is coordinated in a trigonal-pyramidal geometry by the three donor atoms. The P–C bond lengths of the phosphanylmethanide fragment **3** (P–C 174 pm) correspond to those in analogously substituted compounds.[16] This is also the case for the Li–P and Li–N bonds,[17] that do not show any unusual features. As expected, the macrocyclic ring of the Cu^I complex **6**-OTf derived from **4** features the same conformation as **3** (Figure 2). The copper ion, however, forms a "seesaw" arrangement with the two phosphorus and nitrogen atoms. Such a coordination geometry with a quasilinear P-Cu-P (169°) and right-angle N-Cu-N alignment is without precedent in the chemistry of the copper(I) ion[18,19] and possibly explains the observed fluorescence of the complex ($\lambda_{ex} = 400$ nm, $\lambda = 515$ nm). Absorption bands in the region $\lambda = 400$ nm, combined with a shoulder at $\lambda = 455$ nm, are also observed in polarized UV/Vis single-crystal absorption spectra (polarization parallel to the crystalographic *a* axis).[20] On polarizing the incident light perpendicularly to the crystallographic *a* axis the intensity of the absorption band at $\lambda =$ 400 nm distinctly diminishes and the shoulder at $\lambda = 455$ nm almost entirely disappears, which indicates the pronounced anisotropy of the metal-to-ligand charge transfer (MLCT) transitions. UV/Vis absorption spectra of single crystals of the free ligand **4** do not feature any absorption bands in the range 800–350 nm and only at $\lambda \leq 350$ nm display a rise of the absorption curve that could, however, not be resolved in more detail (Figure 3).

Also without precedent is the virtually trigonal-monopyramidal coordination geometry of the nickel center in the complex **7**, where the two phosphorus centers and the CO ligand occupy the equatorial positions (P-Ni-CO 121°, 121°, P-Ni-P 116°; Figure 2). The short P–Ni and the long C–O bonds (172 vs. 117 pm) conform with an effective Ni→CO interaction that is also reflected by the redshift of the CO vibration ($\tilde{\nu} = 1867$ cm^{-1}).[21]

The $\{^1H\}^{31}P$ and $\{^1H\}^7Li$ NMR spectra (25 °C) confirm the coordination of the lithium center to two phosphorus atoms (**2**: $\delta^{31}P = 41.7$ ppm, q; $\delta^7Li = 4.2$ ppm, t, $^1J(P,Li) = 90$ Hz; **3**: $\delta^{31}P = 11.8$ ppm, q; $\delta^7Li = 3.1$ ppm, t, $^1J(P,Li) = 107$ Hz) and consequently the preservation of the ate-complex structure in solution. Intermolecular Li-exchange reactions which would lead to a loss of the ^{31}P–^7Li interaction are not observed between 25 and 60 °C.

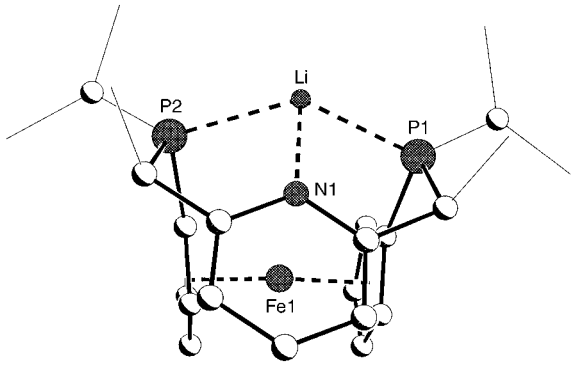

Figure 1. Molecular structure of **3** in the solid state (without H-atoms and peripheral groups). Selected bond lengths [pm] and angles [°]: Li1-N1 203.9(4), Li1-P1 236.5(4), Li1-P2 237.8(4), Li1···Fe1 397.4(4); P1-Li1-P2 128.9(2), N1-Li1-P1 84.7(2), N1-Li1-P2 83.8(2).

Experimental Section

Syntheses of **2, 3**: A suspension of 2,6-di(lithiumbenzyl)pyridine (0.80 g, 1.6 mmol) in THF (20 mL) or 1,1′-dilithioferrocene (0.50 g, 1.6 mmol) in Et_2O/tmeda (10/1; 20 mL) was added dropwise to a solution of bismethylenephosphane **1a** or **1b** (1.0 g 1.6 mmol) in diethyl ether (20 mL) at −78 °C and the mixture stirred for 2 h at this temperature. Upon storage at −30 °C for 2 days the products form as crystalline solids. Yield **2**: 1.72 g (85 %) **3**: 1.26 g (65 %); **3** features a reversible electron transfer: $E^P = 338$ mV (233 K).[22]

4, 5: At 0 °C a solution of **2** (1.0 g, 0.8 mmol) or **3** (1.0 g, 0.9 mmol) in THF (20 mL) was added dropwise to a suspension of triethylamine hydrochloride (0.10 g, 1.8 mmol) in CH_2Cl_2 (30 mL) at 0 °C. Under stirring the reaction solution was allowed to warm to room temperature and then stirred for 2 h. The solvent was removed under reduced pressure and the residue dissolved in diethyl ether (30 mL) to precipitate the lithium chloride which was removed by filtration. The filtrate was concentrated and upon storage at 0 °C for 2–3 days the products crystallize in the form of **4**: colorless **5**: orange crystals. Yield **4**: 0.66 g (92 %) **5**: 0.52 g (71 %); $\{^1H\}^{31}P$ NMR (THF, 25 °C) $\delta =$ 37.7(**4**), $\delta = -15.6$ ppm (**5**); MS (16 eV): m/z (%) = 894 (9.4) $[M]^+$, 636 (100) [M^+–2,6-dibenzylpyridine] (**4**); 813 (53) $[M]^+$, 654 (100) $[M-CH(SiMe_3)_2]^+$ (**5**); **5** features a reversible electron transfer: $E^P =$ 393 mV (298 K).[22]

6-OTf: A solution of **4** (0.30 g, 0.34 mmol) in toluene (15 mL) was added to a suspension of $(CuOTf)_2{\cdot}C_6H_6$ (0.08 g, 0.17 mmol) in toluene (20 mL) at room temperature under stirring. The solution was stirred for 2 h whereupon the product precipitates. After recrystallization from acetonitrile **6** was obtained as yellow to green crystals. Yield: 0.32 g (85 %); $\{^1H\}^{31}P$ NMR (THF, 25 °C): $\delta = 20.7$ ppm; **6** features a reversible electron transfer: $E^P = 970$ mV (298 K).[22]

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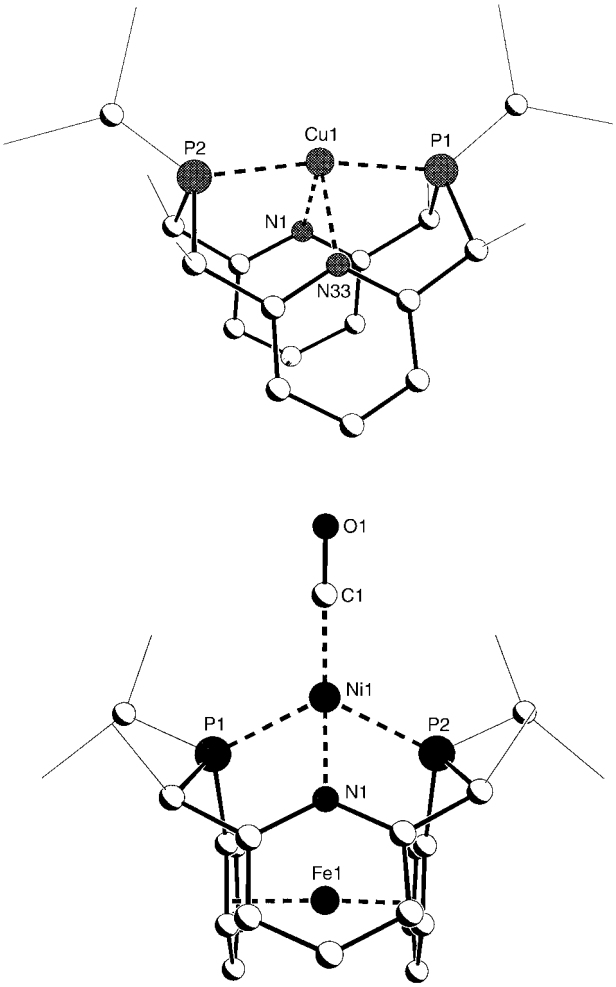

Figure 2. Top: molecular structure of **6** (H atoms and peripheral groups omitted for clarity). Selected bond lengths [pm] and angles [°]: Cu1-N1 214.6(6), Cu1-N33 212.5(6), Cu1-P1 223.8(2), Cu1-P2 223.6(3); N1-Cu1-N33 85.92, N1-Cu1-P1 85.6(2), N1-Cu1-P2 85.9(2), N33-Cu1-P1 85.8(2), N33-Cu1-P2 86.4(2), P1-Cu1-P2 168.9(1), bottom: molecular structure of one independent molecule of **7** in the solid state (H atoms and peripheral groups are omitted for clarity). Selected bond lengths [pm] and angles [°]: Ni1-P1 220.6(1) [220.2(1)], Ni1-P2 221.7(1) [221.1(1)], Ni1-N1 203.1(2) [204.1(2)], Ni1-C1 172.2(3) [172.6(3)], C1-O1 116.6(4) [116.6(4)], Ni1···Fe1 418.9(1) [416.5(1)]; N1-Ni1-P1 81.7(1) [82.3(1)], N1-Ni1-P2 81.6(1) [80.9(1)], N1-Ni1-C1 120.7(1) [121.6(1)], C1-Ni1-P1 121.1(1) [116.0(1)], C1-Ni1-P2 120.8(1) [124.8(1)], P1-Ni1-P2 115.7(1) [116.7(1)].

7: At −30 °C [Ni(CO)$_4$] (0.20 g, 1.17 mmol) was added dropwise to a solution of **5** (0.50 g, 0.61 mmol) in diethyl ether (30 mL) and the solution was stirred for 12 h at room temperature. The excess of [Ni(CO)$_4$] and the solvent were removed under reduced pressure and the residue dissolved in a small amount of diethyl ether. At 0 °C **7** crystallizes within two days in the form of red crystals. Yield: 0.45 g (83 %). MS (16 eV): *m*/*z* (%) 872 (10) [*M*−CO]$^+$, 73 (100) [SiMe$_3$]; {^{1}H}^{31}P NMR (Et$_2$O, 25 °C) $\delta = 32.4$ ppm; $\tilde{\nu} = \nu(\mathrm{CO}) = 1878.6\ \mathrm{cm}^{-1}$; **7** features two reversible electron transfers: $E^{\mathrm{P}} = -556$ and 192 mV (298 K).[22]

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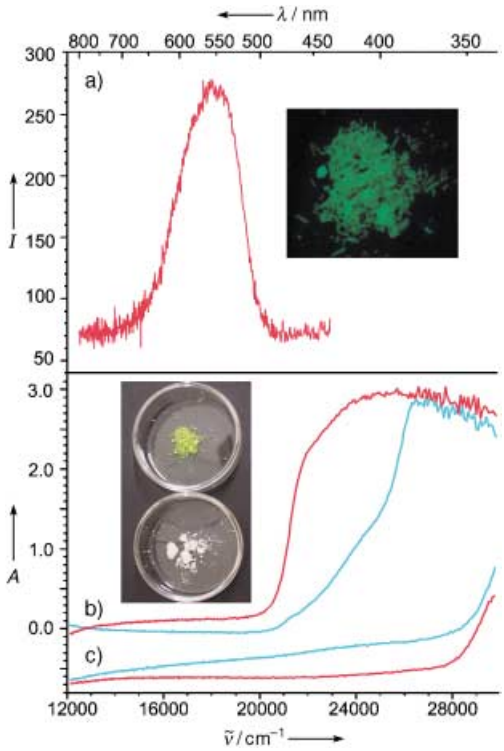

Figure 3. a) Fluorescence spectra of **6**-OTf and photographs of the fluorescing compounds, b) polarized single-crystal UV/Vis spectra of **6** (yellow crystal) and, c) the free ligand **4** (white crystal) on polarization parallel (red spectrum) and perpendicular (blue spectrum) to the *a* axis.

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[10] Synthesis of **1a**: At −78 °C a 1.6 M solution of *n*BuLi in *n*-hexane (4.8 mL, 7.7 mmol) was added to a solution of 2,6-dibenzylpyridine (1.0 g, 3.86 mmol) in THF (30 mL) and tmeda (1.2 mL,

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7.7 mmol). The solution was stirred 0.5 h at −78 °C and then another 5 h at RT. The resulting solution was added dropwise at −78 °C to a solution of ClP=C(SiMe$_3$)$_2$ (1.74 g 7.7 mmol) in diethyl ether (30 mL). After stirring for 1 h at low temperature the solvent was removed under reduced pressure. The residue dissolved in *n*-hexane and then filtrated. After concentration of the filtrate solution **1a** precipitates in crystalline form as isomeric mixture *rac*/*meso*-**1a** = 4:1. MS (16 eV): *m*/*z* (%) = 636 (8) $[M]^+$, 562 (66) $[M-SiMe_3]^+$, 73 (100) $[SiMe_3]^+$; ^{31}P NMR(CH_2Cl_2, 25 °C): δ = 386.2, 388.1 ppm (*rac*/*meso*-**1a**).

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[12] The free L-$N_2P_2[CH(SiMe_3)_2]_2$ ligand **4** prefers a *syn*-chair-chair conformation in the solid state.[8] The ligand **5** retains the conformation of the ate-complex **3**. S. Ekici, M. Nieger, E. Niecke, unpublished results.

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[14] Selected bond lengths [pm] and angles [°] of **2**. Li1-N1 209.7(8), Li1-N33 211.8(7), Li1-P1 244.5(7), Li1-P2 250.1(7); N1-Li1-N33 86.9(3), N1-Li1-P1 87.3(3), N1-Li1-P2 86.9(3), N33-Li1-P1 86.2(3), N33-Li1-P2 87.4(2), P1-Li1-P2 171.6(3). The analogous phosphamethanide-complex bearing four $SiMe_3$ groups instead of the four phenyl groups in **2** has a similar structure.[9]

[15] Crystal data: **3**: $C_{55}H_{115}ClFeLi_3N_7P_2Si_6$, M_r = 1217.14, triclinic, space group $P\bar{1}$ (no. 2), a = 13.6549(1), b = 14.6901(2), c = 19.2675(2) Å, α = 102.043(1), β = 92.848(1), γ = 103.988(1)°, V = 3647.28(7) Å^3, Z = 2, $\mu(Mo_{K\alpha})$ = 0.422 mm^{-1}, $F(000)$ = 1320, 43 173 reflections ($2\theta_{max}$ = 50°), thereof 12 849 unique, $wR2(F^2)$ = 0.1142, $R(F)$ = 0.0406, 670 parameters, 309 restraints. **2**: $C_{72}H_{106}Li_2N_2O_5Si_4$, M_r = 1267.77; orthorhombic, space group $P2_12_12_1$ (no. 19), a = 16.8587(2), b = 20.3906(3), c = 21.2944(3) Å, V = 7320.1(2) Å^3, Z = 4, $\mu(Mo_{K\alpha})$ = 0.173 mm^{-1}, $F(000)$ = 2736, 70 978 reflections ($2\theta_{max.}$ = 50°), thereof 12 972 unique, $wR2(F^2)$ = 0.2342, $R(F)$ = 0.0766, 759 parameters, 505 restraints. The absolute configuration could not be determined reliably (x = 0.40(14)). **6**: $C_{52}H_{68}CuN_2P_2Si_4^+$ $CF_3SO_3^-$, M_r = 1107.99, triclinic, space group $P\bar{1}$ (no. 2), a = 11.2089(5), b = 15.2256(7), c = 18.0123(10) Å, α = 99.324(2), β = 91.194(2), γ = 111.352(2)°, V = 2814.5(2) Å^3, Z = 2, $\mu(Mo_{K\alpha})$ = 0.619 mm^{-1}, $F(000)$ = 1164, 13085 reflections ($2\theta_{max.}$ = 50°), thereof 9316 unique, $wR2(F^2)$ = 0.2148, $R(F)$ = 0.0802, 606 parameters, 581 restraints (disordered $CHTms_2$ groups). **7**: $C_{38}H_{69}NOP_2Si_6FeNi{\cdot}Et_2O$, M_r = 975.10, triclinic, space group $P\bar{1}$ (no. 2), a = 15.0304(1), b = 18.0995(2), c = 20.0055(2) Å, α = 85.709(1), β = 88.418(1), γ = 69.915(1)°, V = 5097.0(1) Å^3, Z = 4, $\mu(Mo_{K\alpha})$ = 0.891 mm^{-1}, $F(000)$ = 2088, 65 240 reflections ($2\theta_{max}$ = 50°), thereof 17 943 unique, $wR2(F^2)$ = 0.1170, $R(F)$ = 0.0424, 986 parameters, 92 restraints (disordered solvent Et_2O). All structure data were collected on a Nonius KappaCCD diffractometer using $Mo_{K\alpha}$-radiation. The structures were solved by direct methods (SHELXS-97) and refined anistropically on F^2, the H-atoms were refined using a riding model (SHELXL-97). CCDC-181615 (**2**), CCDC-181616 (**3**), CCDC-190910 (**6**), and CCDC-190911 (**7**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or deposit @ccdc.cam.ac.uk). SHELXS-97: G. M. Sheldrick, *Acta Crystallogr. Sect. A* **1990**, *46*, 467 – 473 SHELXL-97: G. M. Sheldrick, Universität Göttingen, **1997**.

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[20] The polarized UV/Vis absorption spectra were recorded using a particular micro single-crystal UV/Vis spectrometer. Details about this instrument are prevalent in the literature: E. Krausz, *Aust. J. Chem.* **1993**, *46*, 1041 – 1054. The investigated crystal of compound **6** had a cross-section of 0.20 × 0.08 mm^2, (thickness 0.05 mm), the crystal of compound **4** was notably smaller with a cross-section of 0.10 × 0.05 mm^2 (thickness = 0.05 mm).

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[22] CV: ν = 200 mV s^{-1}; CH_2Cl_2/$n$$Bu_4NBF_4$ 0.1 M; calomel/GCE/Pt (GCE = glassy carbon electrode). MeCN/$n$$Bu_4NPF_6$; reference electrode: calomel E = 0.24 V; IR-compensation (kΩ) **3**: 2.5; **5, 6**: 0.2; **7**: 4.5.

HRMAS NMR Spectroscopy

Application of HRMAS ^{1}H NMR Spectroscopy To Investigate Interactions between Ligands and Synthetic Receptors**

*Heidi Händel, Elke Gesele, Klaus Gottschall, and Klaus Albert**

Dedicated to Professor Günther Jung on the occasion of his 65th birthday

Molecular recognition processes are predicted to become the basis of all advanced separation techniques. The investigation of intermolecular interactions in the interphase between a chromatographic support and a substrate dissolved in a mobile phase is the crucial condition for understanding chromatographic separation mechanisms and for the design of tailored stationary phases. In solution and also in suspension ligand–receptor interactions can be studied using methods of high-resolution (HR) NMR spectroscopy, for example by employing the nuclear Overhauser enhancement (NOE)

[*] Prof. Dr. K. Albert, Dr. H. Händel, Dr. E. Gesele
Institut für Organische Chemie
Universität Tübingen
Auf der Morgenstelle 18, 72076 Tübingen (Germany)
Fax: (+49) 7071-295-875
E-mail: klaus.albert@uni-tuebingen.de
Dr. K. Gottschall
Dr. Gottschall INSTRUCTION
Gesellschaft für Technische Chromatographie mbH
Donnersbergweg 1, 67059 Ludwigshafen (Germany)

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