ELSEVIER

Journal of Organometallic Chemistry 649 (2002) 50–54

Journal of Organometallic Chemistry

www.elsevier.com/locate/jorganchem

Cyclopentadienyl, indenyl, and fluorenyl complexes of sodium with 15-crown-5

Torsten Kähler, Ulrich Behrens, Swen Neander, Falk Olbrich *

Institut für Anorganische und Angewandte Chemie, Universität Hamburg, Martin-Luther-King-Platz 6, D-20146 Hamburg, Germany

Received 21 June 2001; received in revised form 31 October 2001; accepted 11 November 2001

Abstract

The sodium complexes [NaC_5H_5(15-crown-5)] (**1a**), [NaC_9H_7(15-crown-5)] (**1b**), and [$NaC_{13}H_9$(15-crown-5)] (**1c**, C_5H_5 = cyclopentadienyl, C_9H_7 = indenyl, $C_{13}H_9$ = fluorenyl) were synthesized from NaC_5H_5, NaC_9H_7, $NaC_{13}H_9$, and 15-crown-5. Single crystal X-ray diffraction analyses were carried out for all three compounds **1a**, **1b**, **1c**, and show that monomeric units were present in the solid state with the organic aromatic anion coordinated to the sodium cation via the five-membered ring. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Sodium; 15-Crown-5; Cyclopentadienyl; Indenyl; Fluorenyl

1. Introduction

Sodium cyclopentadienide, indenide, and fluorenide are beside the lithium compounds the most important precursors in the organometallic chemistry of the cyclopentadienyl (C_5H_5), indenyl (C_9H_7), and fluorenyl ($C_{13}H_9$) ligand [1]. Due to their unique structural properties, sodium cyclopentadienide and the benzo-anelated analogs have also attracted much interest. In the solid state, base-free NaC_5H_5 forms a polymeric multidecker structure [2] whereas $NaC_{13}H_9$ crystallizes in a salt-like fashion which can be formulated as $Na_2[Na(C_{13}H_9)_3]$ [3]. The solid-state structure of base-free NaC_9H_7 is unknown. Nitrogen- or oxygen-containing Lewis bases are able to break down the polynuclear structures of the above mentioned organo-sodium compounds to aggregates with a lower nuclearity. Mononuclear sodium complexes, for example, have been obtained by the reaction of NaC_9H_7 or $NaC_{13}H_9$ with *N*,*N*,*N*′,*N*″,*N*″-pentamethyldiethylene triamine (PMDTA) yielding the compounds [NaC_9H_7(PMDTA)] [4] and [$NaC_{13}H_9$(PMDTA)] [5]. A monomeric [NaC_5H_5(L)] (L = amine or ether ligand) is still unknown [6], while for lithium the compound [LiC_5H_5(12-crown-4)] was described by Jutzi et al. [7]. Recently we reported on monomeric potassium [8], rubidium, and cesium complexes [9] with the cyclopentadienyl, indenyl, and fluorenyl ligand. These complexes were synthesized by cleaving the polymeric structures of base-free MC_5H_5, MC_9H_7, and $MC_{13}H_9$ (M = K, Rb, Cs) with the crown ether 18-crown-6 yielding the monomeric compounds [MC_5H_5(18-crown-6)], [MC_9H_7(18-crown-6)] and [$MC_{13}H_9$(18-crown-6)].

In this work, we report on the synthesis and structure of monomeric sodium complexes with the macrocyclic Lewis base 15-crown-5.

2. Results and discussion

2.1. Synthesis and NMR spectroscopy

The reaction of NaC_5H_5, NaC_9H_7, and $NaC_{13}H_9$ with 15-crown-5 in tetrahydrofuran yielded the complexes [NaC_5H_5(15-crown-5)] (**1a**), [NaC_9H_7(15-crown-5)] (**1b**), and [$NaC_{13}H_9$(15-crown-5)] (**1c**) as slightly red (**1a**), green (**1b**), and yellow (**1c**) crystalline solids. The yields for the compounds **1a** and **1b** were ca. 80%, for **1c** 20%. All compounds obtained are sensitive against

---

* Corresponding author. Tel.: +49-40-428383112; fax: +49-40-428382893.

E-mail address: olbrich@xray.chemie.uni-hamburg.de (F. Olbrich).

0022-328X/02/$ - see front matter © 2002 Elsevier Science B.V. All rights reserved.

PII: S0022-328X(02)01452-8

Table 1
Crystallographic data for [NaC_5H_5(15-crown-5)] (**1a**), [NaC_9H_7(15-crown-5)] (**1b**) and [$NaC_{13}H_9$(15-crown-5)] (**1c**)

	1a	**1b**	**1c**
Formula	$C_{15}H_{25}NaO_5$	$C_{19}H_{27}NaO_5$	$C_{23}H_{29}NaO_5$
Formula weight	308.34	358.40	408.45
Temperature (K)	173	213	173
Radiation	Mo–K_α	Mo–K_α	Mo–K_α
Crystal size (mm)	0.5 × 0.5 × 0.4	0.4 × 0.4 × 0.3	0.3 × 0.2 × 0.1
Crystal system	Orthorhombic	Orthorhombic	Monoclinic
Space group	*Pbca* (no. 61)	*Pbca* (no. 61)	$P2_1/n$ (no. 14)
Unit cell dimensions			
a (Å)	15.1266(3)	16.758(2)	10.8595(1)
b (Å)	14.5441(1)	12.552(2)	14.6393(2)
c (Å)	15.2965(3)	18.169(3)	13.5875(1)
β (°)			101.537(1)
V (Å^3)	3365.3(1)	3822.0(9)	2116.4(1)
Z	8	8	4
D_{calc} (mg m^{-3})	1.217	1.246	1.282
μ (mm^{-1})	0.111	0.108	0.106
Index ranges	$-19 \leq h \leq 13$ $-18 \leq k \leq 18$ $-18 \leq l \leq 19$	$-21 \leq h \leq 21$ $-16 \leq k \leq 16$ $-23 \leq l \leq 23$	$-14 \leq h \leq 14$ $-17 \leq h \leq 19$ $-8 \leq h \leq 17$
2θ max (°)	55.0	55.0	55.0
Independent reflection	3853	4389	4823
Observed data [$I > 2\sigma(I)$]	2664	3114	3339
Number of parameters	390	235	265
R_1 [$I > 2\sigma(I)$, all]	0.048, 0.080	0.047, 0.072	0.047, 0.082
wR_2 ($I > 2\sigma(I)$, all)	0.090, 0.105	0.116, 0.125	0.092 0.107
GOF	1.100	1.038	1.057
Largest difference peak (e Å^{-3})	0.155/−0.135	0.309/−0.258	0.230/−0.196

air and moisture. They are soluble in tetrahydrofuran, but insoluble in n-hexane.

^{1}H- and ^{13}C-NMR spectroscopy of the new complexes were carried out in THF-d_8. The resonance signal of the methylene protons of the 15-crown-5 ligand was shifted up-field for the complexes **1a** (3.53 ppm), **1b** (3.24 ppm), and **1c** (3.03 ppm) in comparison with the free 15-crown-5 molecule (3.57 ppm). This up-field shift was only 0.04 ppm for complex **1a**, but 0.33 ppm for **1b** and 0.54 ppm for **1c**, and indicated that both the neutral 15-crown-5 and the anionic $C_5H_5^-$, $C_9H_7^-$, and $C_{13}H_9^-$ ligand remained coordinated to the sodium ion in solution. The up-field shift can be explained by the magnetic shielding caused by the benzo-rings present in complex **1b** and **1c**. When mixing equimolar amounts of the complex **1c** with 15-crown-5 a rapid exchange of the coordinated crown ether with free ligand was observed in the ^{1}H-NMR spectrum. Only a single singulett with an averaged value was found at room temperature for the methylene protons of the crown ether.

For the ^{1}H- and ^{13}C-NMR signals of the carbanionic ligands $C_5H_5^-$, $C_9H_7^-$, and $C_{13}H_9^-$ of the complexes **1**, chemical shifts similar to those found for related compounds of the heavier alkali metals were observed [8,9].

2.2. Crystal structures

The solid state structures of the compounds **1a**–**1c** were determined by single crystal X-ray diffraction. Table 1 provides the crystallographic results. For all complexes mononuclear units of the type [NaR(15-crown-5)] (R = C_5H_5, C_9H_7, and $C_{13}H_9$) are observed with almost coplanar 15-crown-5 and R ligands. From one side, the sodium cation is coordinated by the five oxygen atoms of the crown ether, to the other side the anionic aromatic ligand is bound via its five-membered ring (see Figs. 1–3).

A hard sphere electrostatic (HSE) treatment of a contact ion pair between an alkali cation and the indenyl or fluorenyl anion indeed predicts a η^5-coordination of the aromatic ligand to the metal ion [10]. The 15-crown-5 ligand of complex **1a** shows a rotational disorder [11]. The Na–O distances are different within the complexes and vary between 242.0 and 255.2 pm (distances to the disordered 15-crown-5 ligand of complex **1a** not taken into account). The five-membered rings bound to the sodium cation show a rather unsymmetrical coordination mode. For the cyclopentadienyl complex **1a** two close Na–C distances (Na–C4 266.9 pm, Na–C5 270.5 pm), two medium Na–C contacts (Na–C3 284.9 pm, Na–C1 290.5 pm), and one longer

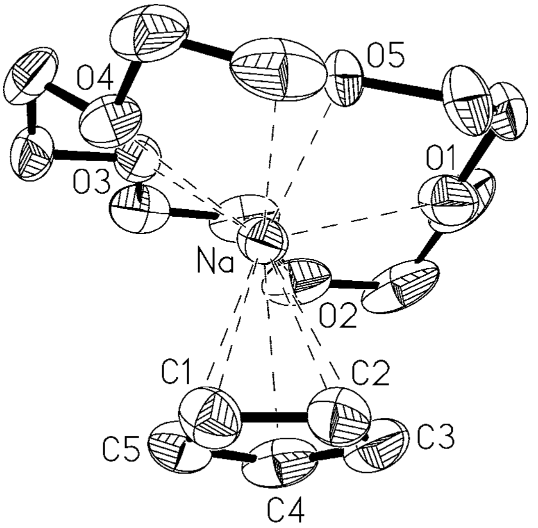

Fig. 1. Molecular structure of [NaC_5H_5(15-crown-5)] (**1a**, H atomes and disorder of the 15-crown-5 ligand are not shown).

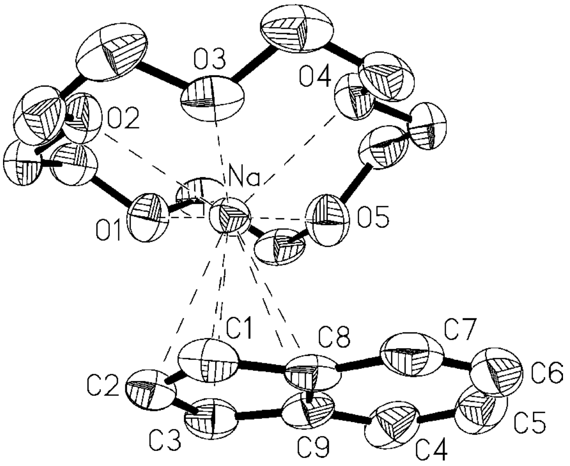

Fig. 2. Molecular structure of [NaC_9H_7(15-crown-5)] (**1b**, H atomes are not shown).

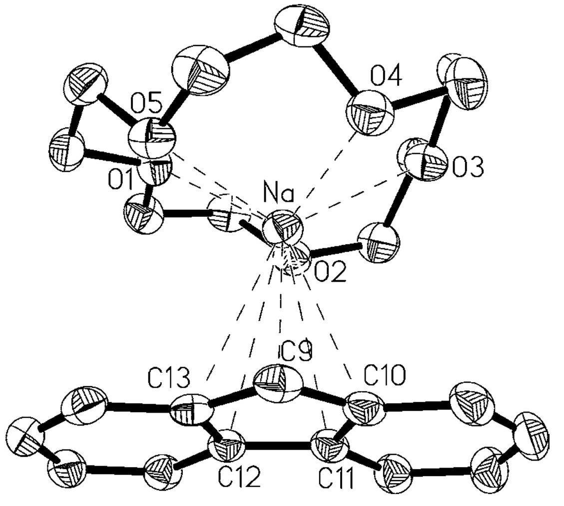

Fig. 3. Molecular structure of [$NaC_{13}H_9$(15-crown-5)] (**1c**, H atomes are not shown).

Na–C distance (Na–C2 299.1 pm) are found. For the indenyl compound **1b** one close Na–C distance (Na–C2 279.3 pm), two medium Na–C contacts (Na–C1 284.4 pm, Na–C3 285.9 pm), and two longer Na–C distances (Na–C8 292.2 pm, Na–C9 292.6 pm) are observed. In the fluorenyl compound **1c** one short Na–C bond to the central most electron rich atom C9 (266.8 pm) is observed, two medium Na–C contacts to the adjacent carbon atoms C10 and C13 (282.0 and 288.8 pm), and two long distances to the carbon atoms C11 and C13 (310.6 and 313.3 pm) are found. The Na–C_{5c} distances (C_{5c} = centroid of the five-membered ring) are 256.4 (**1a**), 260.7 (**1b**), and 266.4 pm (**1c**). Within the aromatic anions cyclopentadienide, indenide, and fluorenide C–C bond lengths are observed as predicted from theoretical calculations [12] (Table 2).

It should be mentioned that in the mononuclear indenyl and fluorenyl complexes [NaC_9H_7(PMDTA)] [4] and [$NaC_{13}H_9$(PMDTA)] [5] (PMDTA = *N,N,N′,N″,N″*-pentamethyldiethylene triamine) shorter Na–C distances to the five-membered ring of the indenyl and fluorenyl ligands are observed, 270.2–279.8 pm for [NaC_9H_7(PMDTA)]; 262.0–304.4 pm for [$NaC_{13}H_9$(PMDTA)]. We think that this effect is due to the smaller coordination number (CN) of the sodium cation in [NaC_9H_7(PMDTA)] and [$NaC_{13}H_9$(PMDTA)] in comparison to **1b** and **1c** (the difference in CN is 2). It should be mentioned further that in the related complexes [$MC_{13}H_9$(18-crown-6)] of the heavier alkali metals K, Rb, and Cs, no η^5-coordination of the fluorenyl ligand has been observed. In the potassium complex [$KC_{13}H_9$(18-crown-6)] [8] the fluorenyl ligand shows an unusual η^6 bonding mode of one six-membered ring and in the complexes [$RbC_{13}H_9$(18-crown-6)] and [$CsC_{13}H_9$(18-crown-6)] [9] the metal ion is located

Table 2

Selected distances (pm) for [NaC_5H_5(15-crown-5)] (**1a**), [NaC_9H_7(15-crown-5)] (**1b**) and [$NaC_{13}H_9$(15-crown-5)] (**1c**)

	1a	**1b**	**1c**
Bond lengths			
Na–O1	– [a]	250.0(1)	242.0(1)
Na–O2	–	250.9(1)	251.0(1)
Na–O3	–	250.1(1)	246.5(1)
Na–O4	–	243.6(1)	247.6(1)
Na–O5	–	246.9(1)	255.2(1)
Na–C1; C1; C9	290.5(2)	284.4(1)	266.8(2)
Na–C2, C2; C10	299.1(2)	279.3(1)	282.0(2)
Na–C3; C3; C11	284.9(2)	285.9(1)	310.6(2)
Na–C4; C8; C12	266.9(2)	292.2(2)	313.3(2)
Na–C5; C9; C13	270.5(2)	292.6(2)	288.8(2)
O–C	– [a]	140.5(2) −143.5(2)	142.1(2) −143.3(2)
C–C [b]	139.3(2) −140.0(2)	136.2(3) −144.9(2)	136.9(3) −144.5(2)

[a] Not given because of disorder.

[b] Anionic ligands.

above the C–C bond between the two adjacent five- and the six-membered rings. It is known that the potential-energy surface above the fluorenyl anion is rather flat, and therefore, steric and crystal packing effects may determine the actual overall solid-state structure of an alkali fluorenyl complex [13].

In the literature, there are described only the crystal structures of two sodium organyls with a crown ether ligand, namely [Na*isodi*Cp(15-crown-5)] (**2**) [13] and [{Na(15-crown-5)}$_2${acenaphthylen(2-)}] (**3**) [15]. Both compounds also have an η^5 coordinated organic ligand and the 15-crown-5 unit.

3. Experimental

3.1. General comments

All manipulations were carried out under purified argon using standard Schlenk techniques. Solvents were distilled under nitrogen from potassium. The crown ether 15-crown-5 was distilled and stored under argon for at least 2 weeks in a Schlenk tube which was connected to a flask containing phosphorus(V)oxide. NaC_5H_5 [2] and NaC_9H_7 [16] were prepared as described in literature. IR spectra, Perkin–Elmer FT-IR 1720; ^{1}H- and ^{13}C-NMR spectra, Varian Gemini 200 BB; X-ray, Siemens SMART CCD and Bruker SMART APEX.

3.2. Synthesis of [NaC$_5$H$_5$(15-crown-5)] (**1a**)

[NaC_5H_5(15-crown-5)] (**1a**) was prepared by adding 1.1 ml (1.22 g, 5.5 mmol) 15-crown-5 to a stirred solution of 0.46 g (5.2 mmol) NaC_5H_5 in 25 ml THF at room temperature (r.t.). A white precipitate was obtained immediately, which was dissolved in 50 ml hot THF. The solution was filtered while still hot and slowly cooled to r.t. Slightly red crystals were formed within several hours. The crystals were separated, washed twice with a small amount (10 ml) of *n*-hexane, and dried for 3 h at 10^{-2} torr. Yield: 1.18 g (73%). Complex **1a** is soluble in THF, but not in *n*-hexane. Melting point (m.p.): 197 °C (dec.). Anal. Calc. Found: for $C_{15}H_{25}NaO_5$: C, 58.4 (58.4); H, 8.2 (8.1); O, 25.9 (23.8)%. IR (Nujol suspension): ν 3072 (s), 2673 (w), 2393 (w), 1694 (w), 1564 (m), 1314 (w), 1294 (w), 1248 (w), 1177 (w), 1030 (s), 1003 (s), 924 (w), 874 (m), 740 (s), 663 (w) cm^{-1}. ^{1}H-NMR (TMS, THF-d_8): δ 3.53 (s, 20H, OCH_2); 5.65 (s, 5H, C_5H_5) ppm. ^{13}C{^{1}H}-NMR (TMS, THF-d_8): δ 68.50 (OCH2); 102.46 (C_5H_5) ppm.

3.3. Synthesis of [NaC$_9$H$_7$(15-crown-5)] (**1b**)

[NaC_9H_7(15-crown-5)] (**1b**) was prepared by adding 0.7 ml (0.78 g, 3.5 mmol) 15-crown-5 to a stirred solution of 0.55 g (2.6 mmol) sodium indenide (NaC_9H_7 containing one mole THF) in 10 ml THF at r.t. Two hours later, the solution was filtered and overlayered with 10 ml *n*-hexane. After several days, the green crystals formed were collected, and washed twice with 10 ml *n*-hexane. Yield: 0.80 g (86%). Complex **1b** is very soluble in THF, but not in *n*-hexane. M.p.: 237 °C (dec.). Anal. Calc. (Found) for $C_{19}H_{27}NaO_5$: C, 63.7 (63.4); H, 7.6 (7.5); O, 22.3 (21.8)%. IR (Nujol suspension): ν 3020 (m), 1967 (w), 1701 (m), 1588 (m), 1555 (m), 1526 (w), 1353 (m), 1323 (w), 1295 (w), 1254 (m), 1218 (w), 1121 (s), 1036 (w), 996 (w), 945 (s), 861 (m), 842 (w), 828 (w), 765 (w), 731 (s), 699 (m), 580 (w), 542 (w), 524 (w), 431 (m) cm^{-1}. ^{1}H-NMR (TMS, THF-d_8): δ 3.24 (s, 20H, OCH_2); 5.85 (d, 2H, 1-, 3-H), 6.29 (m, 2H, 5-, 6-H), 6.51 (t, 1H, 2-H), 7.22 (m, 2H, 4-, 7-H) ppm. ^{13}C{^{1}H}-NMR (TMS, THF-d_8): δ 69.31 (OCH_2); 92.62 (C1/3); 111.89 (C5/6); 116.95 (C2); 119.16 (C4/7); 130.07 (C8/9) ppm.

3.4. Synthesis of [NaC$_{13}$H$_9$(15-crown-5)] (**1c**)

About 0.84 g (5.1 mmol) fluorene were added to a stirred suspension of 0.11 g (4.6 mmol) sodium hydride in 25 ml THF. One hour later, 0.92 ml (1.02 g, 4.6 mmol) 15-crown-5 were added, and the grey suspension was stirred at 50 °C for 2 h. The now dark yellow solution was filtered and overlayered with 20 ml *n*-hexane. After several days, the yellow crystals formed were separated and washed twice with 10 ml *n*-hexane. The mother liquor was evaparated to 15 ml of volume and overlayered again with 20 ml *n*-hexane to increase the yield of **1c**. Total yield: 0.38 g (20%). Complex **1c** is very soluble in THF, but not in *n*-hexane. M.p.: 120 °C (dec.). Anal. Calc. (Found) for $C_{23}H_{29}NaO_5$: C, 67.6 (67.3); H, 7.2 (7.0); O, 19.6 (19.0)%. IR (Nujol suspension): ν 3015 (w), 1656 (w) 1599 (w), 1570 (m), 1354 (m), 1320 (s), 1246 (m), 1220 (s), 1194 (m) 1092 (s), 1000 (m), 982 (m), 948 (s), 866 (m), 842 (w), 827 (w), 748 (s), 722 (s), 664 (w), 574 (m), 523 (w), 493(w), 429 (m) cm^{-1}. ^{1}H-NMR (TMS, THF-d_8): δ 3.03 (s, 20H, OCH_2); 5.99 (s, 1H, 9-H); 6.40 (t, 2H, 3-, 6-H); 6.78 (t, 2H, 2-, 7-H); 7.31 (d, 2H, 1-, 8-H); 7.92 (d, 2H, 4-, 5-H) ppm. ^{13}C{^{1}H}-NMR (TMS, THF-d_8): δ 68.51 (OCH_2); 81.97 (C9); 107.42 (C3/6); 115.92 (C1/8); 118.43 (C4/5); 119.58 (C2/7); 126.49 (C11/12); 136.89 (C10/13) ppm.

3.5. X-ray data collection, structure determination, and refinement

Crystallographic details are provided in Tables 1 and 2. The crystals of the compounds **1a**–**1c** were measured on a CCD area detector diffractometer (Mo–K$_\alpha$ radiation, graphite monochromator, phi and omega-scans). An empirical absorption correction was carried out with the program SADABS [17]. A full hemisphere or sphere of the reciprocal space was scanned with 1271–2550 frames in three to five sets; each frame covered 0.3° in ω. All structures were solved with direct methods (SHELXS-97) [18] and refined with full-matrix least-squares against F_o^2, using the program SHELXL-97 [19]. All non-hydrogen atoms were refined with anisotropic temperature factors. Hydrogen atoms were calculated in idealized positions using a riding model with isotropic temperature factors combined in different logical groups. The 15-crown-5 ligand is disordered in compound **1a**. All atoms were refined with three positions (s.o.f. 0.4, 0.4, and 0.2). C–C and O–C bond lengths were refined with restrained values (SADI option of SHELXL-97). For molecular graphics and publication materials, the program package SHELXTL [20] was used.

4. Conclusions

The macrocyclic ether 15-crown-5 is a useful ligand for sodium organic compounds containing cyclopentadienide, indenide, or fluorenide anions. The polymeric structures of NaC_5H_5 and its benzo-anellated analogs are broken down by 15-crown-5 and mononuclear complexes of the type [NaR(15-crown-5)] ($R = C_5H_5$, C_9H_7, and $C_{13}H_9$) are formed. In these complexes, the sodium ion is sandwiched between the aromatic anion and the crown ether. X-ray structure determinations for all complexes show that the aromatic ligands C_5H_5, C_9H_7, and $C_{13}H_9$ are coordinated to the sodium via their five-membered rings. ^{1}H-NMR investigations prove that dissociation of the ligands from the sodium ion does not occur to a larger portion, but that there is a rapid exchange between the coordinated 15-crown-5 ligand and the free 15-crown-5 molecule in tetrahydrofuran solution.

5. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC nos. 162765 for **1a**, 162766 for **1b**, and 162767 for **1c**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

Acknowledgements

This work was financially supported by the 'Fonds der Chemischen Industrie' and the 'Deutsche Forschungsgemeinschaft', which we gratefully acknowledge (grant Be 760/10-1). F. Olbrich wishes to thank Sachsen-Anhalt for a Habilitation fellowship.

References

[1] W.A. Herrmann, Synthetic Methods of Organometallic and Inorganic Chemistry, vols. 1–8, Georg Thieme Verlag, Stuttgart, 1996/1997.

[2] R.E. Dinnebier, U. Behrens, F. Olbrich, Organometallics 16 (1997) 3855.

[3] R.E. Dinnebier, S. Neander, U. Behrens, F. Olbrich, Organometallics 18 (1999) 2915.

[4] V. Jordan, U. Behrens, F. Olbrich, E. Weiss, J. Organomet. Chem. 517 (1996) 81.

[5] S. Corbelin, J. Kopf, E. Weiss, Chem. Ber. 124 (1991) 2417.

[6] It should be mentioned that there are some monomeric sodium complexes with a substituted cyclopentadienyl ligand: $[NaC_5Me_5(py)_3]$ [21], $[NaC_5HPh_4(dme)_2]$ [22], $[NaC_5HPh_4(thf)_3]$ [23], and $[NaC_5H_3C_5H_8$(15-crown-5)] [14].

[7] H. Chen, P. Jutzi, W. Leffers, M.M. Olmstead, P.P. Power, Organometallics 10 (1991) 1282.

[8] S. Neander, F.E. Tio, R. Buschmann, U. Behrens, F. Olbrich, J. Organomet. Chem. 582 (1999) 58.

[9] S. Neander, U. Behrens, F. Olbrich, J. Organomet. Chem. 604 (2000) 59.

[10] R.J. Bushby, H.L. Steel, M.P. Tytko, J. Chem. Soc. Perkin Trans. 2 (1990) 1155.

[11] Sodium complexes of 15-crown-5 show a motion of the cyclic ether in the solid state: G.W. Buchanan, M. Gerzain, C.I. Ratcliffe, Can. J. Chem. 77 (1999) 1911.

[12] H. Jiao, P. von, R. Schleyer, Y. Mo, M.A. McAllister, T.T. Tidwell, J. Am. Chem. Soc. 119 (1997) 7075.

[13] M. Håkansson, C.-H. Ottosson, A. Boman, D. Johnels, Organometallics 17 (1998) 1208.

[14] J.C. Gallucci, O. Gobley, F. Zaegel, P. Meunier, B. Gautheron, H. Lange, R. Gleiter, N. Kozmina, L.A. Paquette, Organometallics 17 (1998) 111.

[15] H. Bock, C. Arad, C. Näther, J. Organomet. Chem. 520 (1996) 1.

[16] M.L.H. Green, A.K. Hughes, J. Chem. Soc. Dalton Trans. (1992) 527.

[17] G.M. Sheldrick, SADABS: Program for Area Detector Absorption Corrections, University of Göttingen, Germany, 1996.

[18] G.M. Sheldrick, SHELXS-97: Program for Crystal Structure Solution, University of Göttingen, Germany, 1997.

[19] G.M. Sheldrick, SHELXL-97: Program for Crystal Structure Refinement, University of Göttingen, Germany, 1997.

[20] G.M. Sheldrick, SHELXTL, Structure Determination Software Programs, version NT V5.10 (PC), Bruker Analytical X-Ray Instruments, Madison, WI, USA, 2000.

[21] G. Rabe, H.W. Roesky, D. Stalke, F. Pauer, G.M. Sheldrick, J. Organomet. Chem. 403 (1991) 11.

[22] C. Näther, T. Hauck, H. Bock, Acta Crystallogr. Sect. C 52 (1996) 570.

[23] H. Bock, T. Hauck, C. Näther, Z. Havlas, Z. Naturforsch. Teil. B 52 (1997) 524.