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The preparation and crystal structures of sodium and potassium pentamethylcyclopentadienyl pyridine solvates

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Abstract

The compounds $(C_5Me_5)Na \cdot 3py$ (**1**) and $(C_5Me_5)K \cdot 2py$ (**2**) have been prepared and their crystal structures determined at low temperatures. The crystal structure of **1** exhibits a three-legged stool monomer of composition $(C_5Me_5)Na \cdot 3py$, whereas **2** has a linear zig-zag chain structure of composition $[(C_5Me_5)K \cdot 2py]_\infty$.

Introduction

The alkali metal salts of pentamethylcyclopentadiene (Cp*H) are important reagents for attaching Cp*-groups to metal complexes. The presence of Cp*-ligands [1] enhances the solubility of transition metal complexes in organic solvents and has also allowed the isolation of Main Group metal compounds in formally low oxidation states [2].

In recent years there has been a growing interest in the solid state structures of alkali metal cyclopentadiene derivatives, but relatively few stable crystalline alkali metal salts of cyclopentadiene derivatives have been isolated and their structures determined. The crystal structures of $[C_5H_4(SiMe_3)]Li \cdot TMEDA$ [3], $[C_5H_2(SiMe_3)_3]Li \cdot TMEDA$ [4], $[C_5H_2(SiMe_3)_3]Li \cdot$ Chinuclidin [4], $[C_5H_2(SiMe_3)_3]Li \cdot PMDETA$ [5], $(C_5H_4Me)Li \cdot TMEDA$ [6], $(C_5H_5)Na \cdot TMEDA$ [7], $(C_5H_4COMe)Na \cdot THF$ [8], $[C_5H_4(SiMe_3)]K$ [9] and $[C_5(CH_2C_6H_5)_5]K \cdot 3THF$ [10] are known. It is interesting that no structures of alkali metal Cp* salts have been reported. We describe here the preparation and structural determination of the two new alkali metal Cp* salts $(C_5Me_5)Na \cdot 3py$ (**1**) and $(C_5Me_5)K \cdot 2py$ (**2**).

Results and discussion

Pentamethylcyclopentadiene reacts cleanly with sodium metal and potassium hydride to give the corresponding alkali metal salts in good yield according to

Table 1

Crystallographic data for **1** and **2**

Formula	$C_{25}H_{30}N_3Na$	$C_{20}H_{25}N_2K$
Formula weight	395.5	332.5
Data collection at T (°C)	−120	−120
Crystal size (mm)	0.1×0.2×0.4	0.4×0.4×0.6
Space group	*Pbca*	$P2_1/c$
a (Å)	15.271(1)	11.454(1)
b (Å)	17.456(1)	10.393(2)
c (Å)	17.680(2)	17.252(3)
β (°)	90	106.93(1)
Cell volume (Å^3)	4713	1965
Formula units per cell Z	8	4
Calculated density ρ (M gm^{-3})	1.115	1.124
Absorption coefficient μ (mm^{-1})	0.077	0.27
$F(000)$	1696	712
Measured 2θ-range (°)	8–45	8–50
Number of measured reflections	3126	3983
Number of unique reflections	3058	3454
Number of observed reflections	1666	2816
Consistency of equivalent reflections (R_{int})	0.0093	0.0547
$F > p\sigma(F)$; $p =$	3	3
R	0.0666	0.0427
wR	0.0608	0.0496
Goodness of fit	1.73	1.59
Weighting factor g $w = 1/(\sigma^2(F) + \mathrm{abs}(g)F^2)$	0.0003	0.0004
Refined parameters	262	283
Last difference-Fourier synthesis		
Highest maximum (e$^-$Å^{-3})	0.26	0.25
Lowest minimum (e$^-$Å^{-3})	0.32	0.23

equations 1 and 2.

$$\mathrm{Na} + \mathrm{C_5Me_5H} \xrightarrow[-1/2\mathrm{H_2}]{\text{Toluene}} \mathrm{NaC_5Me_5} \qquad (1)$$

$$\mathrm{KH} + \mathrm{C_5Me_5H} \xrightarrow[-\mathrm{H_2}]{\text{THF}} \mathrm{KC_5Me_5} \qquad (2)$$

By dissolving the products from reactions 1 and 2 in pyridine and slowly removing the solvent, single crystals of **1** and **2** suitable for X-ray structural analyses were obtained. Crystal data for **1** and **2** are given in Table 1, fractional coordinates in Tables 2 (**1**) and 3 (**2**), and selected interatomic distances and angles in Tables 4 (**1**) and 5 (**2**).

The crystal structure of **1** consists of a three-legged stool monomer with the $\mathrm{Cp}^{\star -}$ pentahapto coordinated to sodium together with three molecules of pyridine (Fig. 1). The molecular structure of $(\mathrm{C_5Me_5})\mathrm{Na} \cdot 3\mathrm{py}$ shows it to be the first example of a monomeric cyclopentadienyl derivative of sodium. The Na–C bond distances (mean

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* A reference number with an asterisk indicates a note in the list of references.

Table 2

Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients [a] ($Å^2 \times 10^3$) for **1**

	x	y	z	U_{eq}
Na(1)	−32(1)	1171(1)	1603(1)	43(1)
C(1)	−382(3)	2582(3)	1117(3)	43(2)
C(2)	−189(3)	2660(2)	1885(3)	43(2)
C(3)	716(4)	2506(3)	1972(3)	44(2)
C(4)	1058(3)	2336(3)	1250(3)	47(2)
C(5)	379(4)	2385(3)	735(3)	47(2)
C(6)	−1265(4)	2748(3)	767(3)	78(3)
C(7)	−813(4)	2917(3)	2499(3)	75(2)
C(8)	1230(4)	2563(3)	2700(3)	78(3)
C(9)	1991(4)	2119(3)	1053(3)	78(3)
C(10)	456(4)	2268(3)	−114(3)	79(3)
N(1)	1021(3)	161(2)	1283(2)	49(2)
C(11)	1663(4)	202(3)	1790(3)	58(2)
C(12)	2341(4)	−308(3)	1854(3)	59(2)
C(13)	2377(4)	−910(3)	1351(3)	58(2)
C(14)	1732(4)	−964(3)	825(3)	54(2)
C(15)	1080(4)	−428(3)	799(3)	50(2)
N(2)	−1399(3)	529(3)	1252(2)	54(2)
C(16)	−1435(4)	−212(3)	1081(3)	50(2)
C(17)	−2205(4)	−602(3)	957(3)	60(2)
C(18)	−2979(4)	−212(4)	1017(3)	65(3)
C(19)	−2968(4)	550(4)	1188(3)	67(3)
C(20)	−2161(5)	887(3)	1301(3)	62(2)
N(3)	−225(3)	580(2)	2873(2)	50(2)
C(21)	−485(4)	983(3)	3458(3)	66(2)
C(22)	−731(5)	665(3)	4132(3)	87(3)
C(23)	−687(4)	−116(3)	4225(3)	63(2)
C(24)	−413(3)	−550(3)	3634(3)	45(2)
C(25)	−195(3)	−175(3)	2970(3)	49(2)

[a] Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

2.68 Å) in **1** are considerably shorter than those found in $(C_5H_5)Na \cdot TMEDA$ [7] (2.92 Å) and $(C_5H_4COMe)Na \cdot THF$ [8] (2.82 Å).

The Na–Cp(centre) distance is 2.40 Å. The mean bond length between the sodium and the pyridine N-atoms is 2.46 Å. The ^{23}Na NMR spectrum of $(C_5Me_5)Na$ in C_5D_5N solution gives a single broad line ($\Delta\nu_{1/2} = 420$ Hz) at −22.0 ppm [11*] (Fig. 2). Presumably the relatively large line width arises from large quadrupolar relaxation effects owing to a highly asymmetric environment around the sodium nucleus [12]. In general ^{23}Na NMR line widths are of the order of a few Hz for solvated sodium cations [13]. The asymmetric environment of sodium in **1** is probably due to the presence of strong contact $Cp^{\star -}Na^+$ ion pairs in solution. Furthermore, it is likely that $(C_5Me_5)Na \cdot 3py$ retains its monomeric structure in pyridine solution.

In contrast to **1**, the crystal structure of **2** consists of polymeric zig-zag chains of composition $[(C_5Me_5)K \cdot 2py]_\infty$ (Fig. 3). In addition to two pyridine molecules, each potassium cation in the chain is approximately pentahapto coordinated to two Cp*-rings. The angle between the cyclopentadienyl ring normals at the potassium is 138°, smaller than the corresponding angle in the analogous polymer of

Table 3

Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients [a] ($Å^2 \times 10^3$) for **2**

	x	y	z	U_{eq}
K(1)	4093(1)	178(1)	7365(1)	30(1)
C(10)	4885(2)	−2362(2)	6777(1)	32(1)
C(1)	4978(3)	−2390(3)	5924(2)	56(1)
C(20)	3884(2)	−2761(2)	7033(1)	33(1)
C(2)	2668(2)	−3215(3)	6490(2)	55(1)
C(30)	4205(2)	−2640(2)	7886(1)	31(1)
C(3)	3436(3)	−2985(3)	8430(2)	51(1)
C(40)	5404(2)	−2152(2)	8152(1)	29(1)
C(4)	6065(3)	−1784(2)	9012(1)	48(1)
C(50)	5825(2)	−1987(2)	7469(1)	29(1)
C(5)	7077(2)	−1538(2)	7477(2)	45(1)
N(1)	2287(2)	143(2)	8227(1)	46(1)
C(11)	2336(3)	221(3)	9003(2)	58(1)
C(12)	1371(3)	−12(3)	9299(2)	57(1)
C(13)	290(3)	−350(3)	8776(2)	63(1)
C(14)	220(3)	−443(5)	7977(2)	100(2)
C(15)	1237(3)	−211(4)	7735(2)	73(1)
N(2)	1955(2)	490(2)	5906(1)	49(1)
C(21)	2361(2)	168(3)	5290(2)	56(1)
C(22)	1887(3)	638(3)	4511(2)	65(1)
C(23)	926(3)	1478(3)	4365(2)	66(1)
C(24)	486(2)	1809(3)	4982(2)	58(1)
C(25)	1020(2)	1297(3)	5740(2)	50(1)

[a] Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 4

Selected bond lengths (Å) and angles (°) for **1**

Na(1)–C(1)	2.663(5)	Na(1)–N(3)	2.488(4)
Na(1)–C(2)	2.658(5)	C(1)–C(2)	1.397(7)
Na(1)–C(3)	2.676(5)	C(1)–C(5)	1.387(8)
Na(1)–C(4)	2.701(5)	C(2)–C(3)	1.416(8)
Na(1)–C(5)	2.691(5)	C(3)–C(4)	1.410(7)
Na(1)–N(1)	2.451(5)	C(4)–C(5)	1.382(8)
Na(1)–N(2)	2.450(5)	Na(1)–Cp*(centre)	2.399(−)
C(1)–Na(1)–C(2)	30.4(1)	C(1)–Na(1)–N(2)	99.8(2)
C(1)–Na(1)–C(3)	50.2(2)	C(2)–Na(1)–N(2)	114.7(2)
C(2)–Na(1)–C(3)	30.8(2)	C(3)–Na(1)–N(2)	145.5(2)
C(1)–Na(1)–C(4)	49.6(2)	C(4)–Na(1)–N(2)	144.2(2)
C(2)–Na(1)–C(4)	50.4(2)	C(5)–Na(1)–N(2)	114.5(2)
C(3)–Na(1)–C(4)	30.4(2)	N(1)–Na(1)–N(2)	99.9(2)
C(1)–Na(1)–C(5)	30.0(2)	C(1)–Na(1)–N(3)	130.6(2)
C(2)–Na(1)–C(5)	50.0(1)	C(2)–Na(1)–N(3)	103.0(2)
C(3)–Na(1)–C(5)	49.7(1)	C(3)–Na(1)–N(3)	101.0(1)
C(4)–Na(1)–C(5)	29.7(2)	C(4)–Na(1)–N(3)	126.4(2)
C(1)–Na(1)–N(1)	136.2(2)	C(5)–Na(1)–N(3)	150.3(2)
C(2)–Na(1)–N(1)	143.7(2)	N(1)–Na(1)–N(3)	89.3(1)
C(3)–Na(1)–N(1)	113.7(2)	N(2)–Na(1)–N(3)	86.4(1)
C(4)–Na(1)–N(1)	94.9(2)		
C(5)–Na(1)–N(1)	106.4(2)		

Table 5

Selected bond lengths (Å) and angles (°) for **2**

K(1)-C(10)	3.059(2)	K(1)-C(20)	3.104(2)
K(1)-C(30)	3.055(2)	K(1)-C(40)	2.962(2)
K(1)-C(50)	2.970(2)	K(1)-N(1)	2.879(2)
K(1)-N(2)	2.974(2)	K(1)-C(10A)	3.014(2)
K(1)-C(20A)	3.104(2)	K(1)-C(30A)	3.102(2)
K(1)-C(40A)	3.020(2)	K(1)-C(50A)	2.959(2)
C(10)-C(20)	1.406(3)	C(10)-C(50)	1.409(3)
C(20)-C(30)	1.415(3)	C(30)-C(40)	1.409(3)
C(40)-C(50)	1.407(3)		
C(10)-K(1)-C(20)	26.4(1)	C(10)-K(1)-C(30)	43.9(1)
C(20)-K(1)-C(30)	26.6(1)	C(10)-K(1)-C(40)	44.5(1)
C(20)-K(1)-C(40)	44.1(1)	C(30)-K(1)-C(40)	27.0(1)
C(10)-K(1)-C(50)	27.0(1)	C(20)-K(1)-C(50)	44.0(1)
C(30)-K(1)-C(50)	44.4(1)	C(40)-K(1)-C(50)	27.4(1)
C(10)-K(1)-N(1)	119.2(1)	C(20)-K(1)-N(1)	93.1(1)
C(30)-K(1)-N(1)	79.1(1)	C(40)-K(1)-N(1)	95.6(1)
C(50)-K(1)-N(1)	121.8(1)	C(10)-K(1)-N(2)	93.7(1)
C(20)-K(1)-N(2)	87.2(1)	C(30)-K(1)-N(2)	107.9(1)
C(40)-K(1)-N(2)	131.3(1)	C(50)-K(1)-N(2)	120.3(1)
N(1)-K(1)-N(2)	84.3(1)	C(10)-K(1)-C(10A)	141.7(1)
C(20)-K(1)-C(10A)	157.4(1)	C(30)-K(1)-C(10A)	133.7(1)
C(40)-K(1)-C(10A)	113.3(1)	C(50)-K(1)-C(10A)	117.1(1)
N(1)-K(1)-C(10A)	89.5(1)	N(2)-K(1)-C(10A)	115.4(1)
C(10)-K(1)-C(20A)	116.2(1)	C(20)-K(1)-C(20A)	138.7(1)
C(30)-K(1)-C(20A)	126.8(1)	C(40)-K(1)-C(20A)	100.4(1)
C(50)-K(1)-C(20A)	94.7(1)	N(1)-K(1)-C(20A)	114.5(1)
N(2)-K(1)-C(20A)	124.0(1)	C(10A)-K(1)-C(20A)	26.5(1)
C(10)-K(1)-C(30A)	109.0(1)	C(20)-K(1)-C(30A)	135.4(1)
C(30)-K(1)-C(30A)	140.1(1)	C(40)-K(1)-C(30A)	114.0(1)
C(50)-K(1)-C(30A)	97.3(1)	N(1)-K(1)-C(30A)	131.0(1)
N(2)-K(1)-C(30A)	101.4(1)	C(10A)-K(1)-C(30A)	43.8(1)
C(20A)-K(1)-C(30A)	26.4(1)	C(10)-K(1)-C(40A)	126.5(1)
C(20)-K(1)-C(40A)	149.4(1)	C(30)-K(1)-C(40A)	166.4(1)
C(40)-K(1)-C(40A)	140.5(1)	C(50)-K(1)-C(40A)	122.2(1)
N(1)-K(1)-C(40A)	113.0(1)	N(2)-K(1)-C(40A)	80.3(1)
C(10A)-K(1)-C(40A)	44.4(1)	C(20A)-K(1)-C(40A)	43.7(1)
C(30A)-K(1)-C(40A)	26.6(1)	C(10)-K(1)-C(50A)	152.6(1)
C(20)-K(1)-C(50A)	175.0(1)	C(30)-K(1)-C(50A)	158.2(1)
C(40)-K(1)-C(50A)	140.5(1)	C(50)-K(1)-C(50A)	138.3(1)
N(1)-K(1)-C(50A)	88.2(1)	N(2)-K(1)-C(50A)	88.2(1)
C(10A)-K(1)-C(50A)	27.3(1)	C(20A)-K(1)-C(50A)	44.0(1)
C(30A)-K(1)-C(50A)	44.1(1)	C(40A)-K(1)-C(50A)	27.2(1)
K(1)-C(10)-K(1A)	132.8(1)	K(1)-C(20)-K(1A)	127.4(1)
K(1)-C(30)-K(1A)	129.3(1)	K(1)-C(40)-K(1A)	136.9(1)
K(1)-C(50)-K(1A)	139.6(1)		

$[C_5H_4(SiMe_3)]K$ (150°). This is presumably due to the additional coordination of two pyridine molecules to the potassium in **2**. The K–Cp(centre) distances (mean 2.79 Å) are similar to the corresponding distances found in $[(C_5H_4(SiMe_3)]K$ (2.78 Å) and in the monomeric $[C_5(CH_2C_6H_5)_5]K \cdot 3THF$ (2.79 Å). The K–C distances (mean 3.03 Å) are comparable to those in $[(C_5H_4(SiMe_3)]K$ (3.03 Å) and in

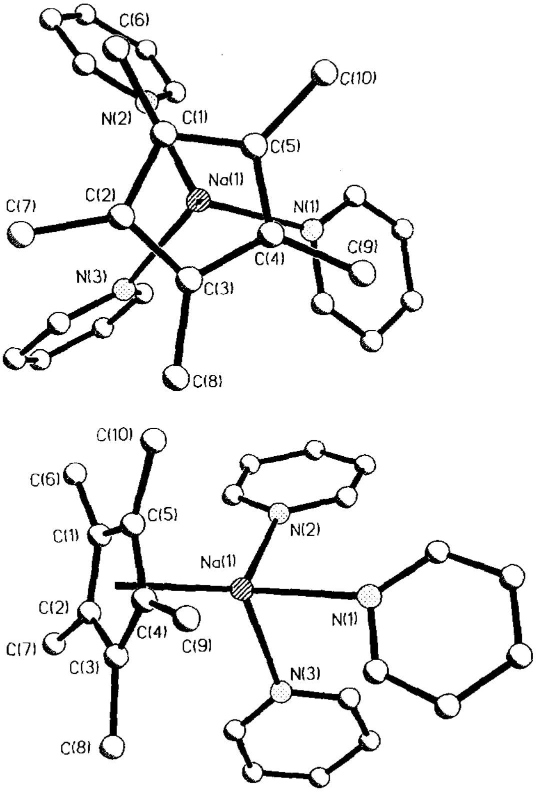

Fig. 1. Two views of the molecular structure of the $[C_5Me_5]Na \cdot 3py$ monomer of 1.

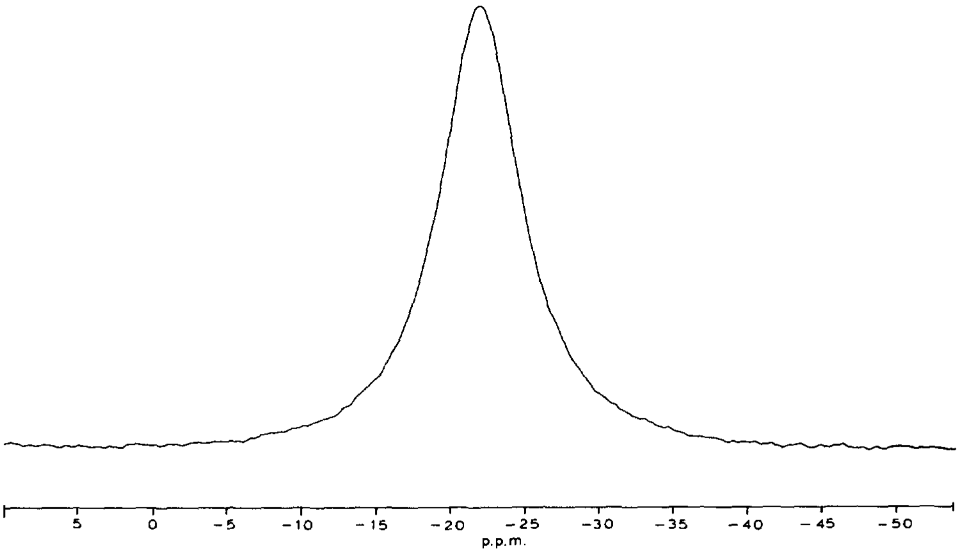

Fig. 2. Solution ^{23}Na-NMR spectrum of $(C_5Me_5)Na$ in C_5D_5N at 25° C.

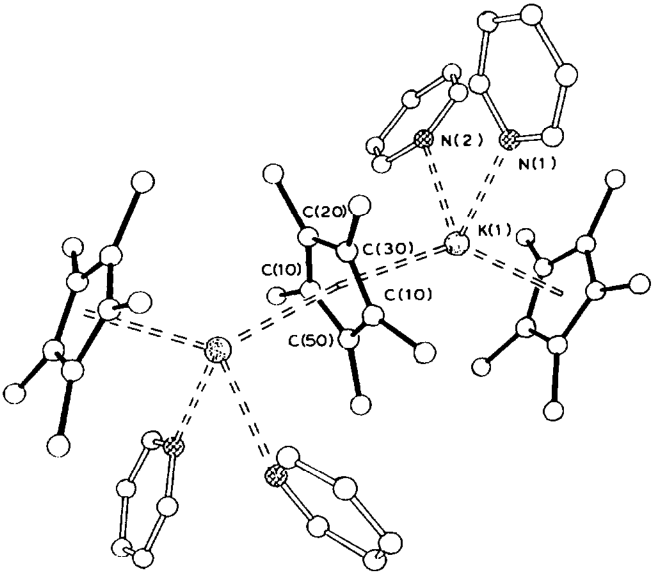

Fig. 3. The structure of the zig-zag $[(C_5Me_5)K \cdot 2py]_\infty$ chain of **2**.

$[C_5(CH_2C_6H_5)_5]K \cdot 3THF$ (3.04 Å). The K–N distances in **2** are 2.879(2) and 2.974(2) Å.

The change in structure from monomeric to polymeric for **1** and **2** and the increasing coordination number at the metal can be accounted for in terms of the increasing alkali metal radius. Consequently by substituting the sodium in the $(C_5Me_5)Na \cdot 3py$ monomer with a larger and less polarizing potassium would result in decreased steric crowding and weaker $Cp^{\star -}M^+$ ion pair and cation-ligand interactions. Thus removal of one pyridine ligand from a $(C_5Me_5)K \cdot 3py$ monomer would allow a second $Cp^\star$-ring to coordinate to the potassium with resulting chain formation.

Experimental

All syntheses were carried out under pure nitrogen in dried and freshly distilled solvents. Mass spectrum: Finnigan 8230. NMR-spectra in C_5D_5N (standards): ^1H, ^{13}C ($TMS_{ext.}$); ^{23}Na ($NaCl_{sat.}$ in D_2O, $\Delta\nu_{1/2} = 13$ Hz at 25 °C) [11*]: Bruker AM 250.

Sodium pentamethylcyclopentadiene

Sodium metal and a solution of (1.0g, 43 mmol) pentamethylcyclopentadiene (7.0g, 51 mmol), in toluene (80 ml) were vigorously stirred together for 3 d under reflux. After filtration and washing with toluene (30 ml) the residue was extracted with tetrahydrofuran. Removal of the solvent gave 6.5 g (95%) of sodium pentamethylcyclopentadiene as a white powder; m.p. > 250 °C. ^1H NMR (C_5D_5N): $\delta(Me) = 2.41$ ppm; ^{13}C NMR (C_5D_5N): $\delta(Me) = 12.4$, (C_5) 105.5 ppm; ^{23}Na NMR (C_5D_5N): $\delta(Na) = -22.0$ ppm.

Potassium pentamethylcyclopentadiene

A mixture of potassium hydride (1.0 g, 25 mmol), pentamethylcyclopentadiene (4.0g, 29 mmol), and tetrahydrofuran (40 ml) was stirred for 14 h at room temperature then filtered. Washing of the residue with tetrahydrofuran (30 ml) and drying under vacuum gave 4.1 g (95%) of potassium pentamethylcyclopentadiene as a white powder; m.p. > 250 °C. ^1H NMR (C_5D_5N): $\delta(Me) = 2.23$ ppm; ^{13}C NMR (C_5D_5N): $\delta(Me) = 11.91$, (C_5) 106.1 ppm; MS (FD): m/z (ion) = 213 ($M + K^+$), 39 (K^+).

Crystal growth and X-ray measurements of 1 and 2

Crystals of **1** and **2** were grown during 1–2 weeks by slowly condensing the solvent off a pyridine solution of $(C_5Me_5)Na$ or $(C_5Me_5)K$.

Both structures were measured on a Stoe–Siemens four-circle diffractometer with graphite-monochromated Mo-K_α radiation ($\lambda = 71.069$ pm). Data were collected at −120 °C with a profile fitting method [14], $2\theta = 8$–$45°$ (**1**) and 8–50° (**2**). The structures were solved by Patterson methods and refined by full-matrix least squares, with all non-hydrogen atoms anisotropic. All hydrogen atoms were found by difference Fourier synthesis and refined isotropically. A riding model was employed for the hydrogen atoms with an idealized bond length of 96 pm. A semi-empirical absorption correction was applied in both cases. All calculations were performed with SHELX-76 and SHELXS-86 [15,16].

Supplementary material available. Tables of intensity measurement and refinement parameters, bond lengths, bond angles, anisotropic displacement parameters, H atom coordinates and isotropic displacement parameters and a list of observed and calculated structural factors are available from the authors. Further details of the X-ray structural investigation can be obtained from the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, D-7514 Eggenstein-Leopoldshafen 2, under quotation of the deposit number CSD-54823, the authors and the publication.

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