Organometallics **1999,** *18,* 2915−2918

Solid-State Structures of Base-Free Indenyllithium and Fluorenylsodium

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Received February 17, 1999

Summary: The solid-state structures of two important organometallic reagents, base-free indenyllithium and base-free fluorenylsodium, were determined by high-resolution X-ray powder diffraction experiments. Indenyllithium crystallizes as an infinite chain in the fashion of a "multidecker structure", whereas fluorenylsodium forms the saltlike structure $Na_2[NaFl_3]$, containing the novel trigonal $[NaFl_3]^{2-}$ anion.

Introduction

Lithium organometallic compounds are of special interest to organometallic chemists because of their widespread use in chemical laboratories and in industry applications.[1−3] Since the solid-state structures of the fundamental compounds $[LiMe]_4$ and $[LiEt]_4$ have been determined by X-ray diffraction experiments,[4,5] few structures of binary organolithium compounds (compounds formed between lithium cations and hydrocarbon anions without other neutral ligands) have been reported. Several years ago, Stalke et al. were able to solve the single-crystal structures of the alkyllithium compounds $[Li^nBu]_6$ and $[Li^tBu]_4$.[6] The structures of binary aryllithium compounds were determined from LiPh[7] and from several lithium compounds of phenyl derivatives with bulky substituents, i.e., $[LiC_6H_3Mes_2]_2$,[8] $[LiC_6H_3\text{-}3,5\text{-}^tBu_2]_6$,[9] and $[LiC_6H_2(^iPr)_3]_4$.[8] Structures of binary lithium organometallic compounds with π-bonding anions are only known for LiCp,[10] LiCp*,[11] $[LiFl]_2$[12] (Fl = fluorenyl), and one example of an unsolvated vinyllithium derivative ($[LiCPh{=}C_9H_4Me_4]_3$),[13] respectively. It is worth mentioning that the structures of $[LiMe]_4$ and all base-free polymeric lithiumorganic compounds were solved by powder diffraction methods. Even fewer structural reports can be found in the literature for binary sodium organometallic compounds. Here, the examples $[NaMe]_4$,[14] NaEt,[15] and NaCp[10] are given. Drawing on our systematic investigations of the solid-state structures of base-free organo alkali metal compounds, we report on new crystal structures for base-free indenyllithium, a missing link in the series of π-bonding anions in organometallic chemistry, and on the structure of fluorenylsodium, a new example of a base-free sodium organometallic compound. The structures of both compounds have been investigated by high-resolution X-ray powder diffraction methods.

Results and Discussion

Base-free indenyllithium (**1**) was obtained as a white microcrystalline powder by the reaction of indene with *n*-butyllithium in diethyl ether in a procedure similar to those described in previous articles.[16] The reaction of sodium hydride with fluorene in a mixture of tetrahydrofuran and toluene yielded yellow crystals of the polymeric compound $[NaFl(thf)_2]_n$ (**3**).[17] The tetrahydrofuran ligands could be easily removed by stirring **3** in *n*-hexane at room temperature for several hours, to

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[17] The structures of the complexes $[NaFl(thf)_2]_n$, $[(KFl)_2thf]_n$, and $[(CsFl)_2thf]_n$ were investigated by single-crystal structure analysis. The compounds will be subject of a separate, upcoming publication. Crystallographic data are already available at the Cambridge Crystallographic Database (CCDC-114096 ($[NaFl(thf)_2]_n$), CCDC-114093 ($[(KFl)_2thf]_n$), and CCDC-114094 ($[(CsFl)_2thf]_n$)).

10.1021/om990108r CCC: $18.00
Publication on Web 06/22/1999

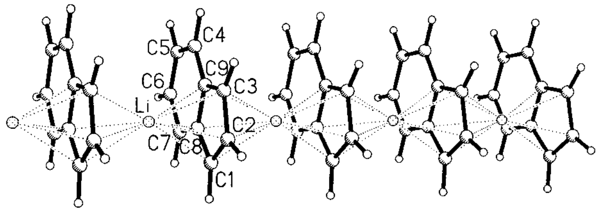

Figure 1. Polymeric chain of LiInd (**1**, five LiInd units shown). Distances (Å): Li−C(1) 2.318(3); Li−C(2) 2.289(3); Li−C(3) 2.304(3); Li−C(8) 2.350(3); Li−C(9) 2.341(3); Li−$C5_{centroid}$ 1.983; C···C 1.416(1) (equal restrained distances for all bonds within the indenyl ring). Angles (deg): Li′−Li−Li″ 180.0; $C5_{centroid}$−Li−$C5_{centroid}$′ 176.5.

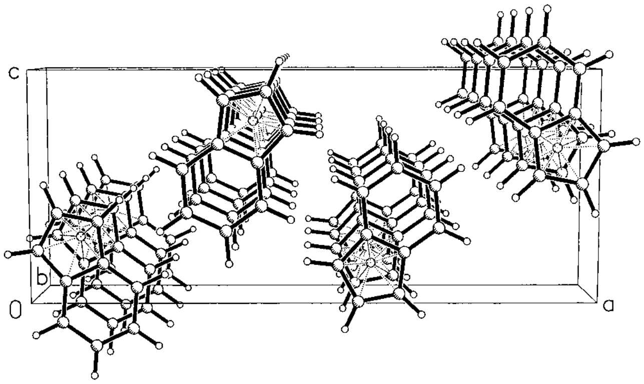

Figure 2. Drawing of the unit cell of LiInd (**1**): View along the *b*-axis.

give a yellow microcrystalline powder of base-free fluorenylsodium (**2**). The compounds **1** and **2** are highly sensitive to moisture and air. However, they can be stored under argon for weeks without decomposition. Although we always found a small amount of fluorene in our NaFl samples, even after experimenting with different modifications in synthesis and isolation procedures, this did not interfere with our crystallographic investigations of **2** (see X-ray crystallography). Similar reactions were carried out with the heavier alkali metals potassium, rubidium, and cesium. We obtained the THF adducts $[(MFl)_2thf]_n$ (M = K, Cs).[17] Unfortunately, the bonding of the THF ligand to the metal atoms in these complexes was so strong that a complete removal of the tetrahydrofuran molecules was not possible.

Indenyllithium crystallizes as an infinite chain in the fashion of a "multidecker structure". The Li^+ ions are linear η^5 coordinated by the five-membered rings (C5-rings) of two indenyl anions (Figure 1) and located above the center of the C5-rings (angle $C5_{centroid}$−Li−$C5_{centroid}$′ 176.5°). They are not shifted toward the six-membered ring (C6-ring). The chain of the lithium cations is linear and parallel to the crystallographic *b*-direction (Li′−Li−Li″ 180°; mirror plane perpendicular to the short *b*-axis). Within the *ac* plane the lithium cations are surrounded by four indenyl anions (Figure 2), but bonding interactions to these anions are not observed.

When the structure of **1** is compared with the structures of the related compounds LiCp,[10] LiCp*,[11] and $[LiFl]_2$,[12] striking similarities to LiCp and LiCp* are found but not to $[LiFl]_2$. In fact, the three compounds LiInd, LiCp, and LiCp* crystallize in the same polymeric fashion, with two C5-rings linear η^5 bonded to the Li^+ ions. On the other hand, in fluorenyllithium, the Li^+ ions prefer the linear coordination of two η^6 bonded C6-rings of two Fl^- anions and build up a sandwich-shaped binuclear molecule instead of a polymeric chain. For the isostructural compounds LiInd, LiCp, and LiCp*, a comparison of the Li−C distances shows the following order within the three anions: Cp* < Cp < Ind (Li−C bond lengths (average): LiCp* 2.255 Å; LiCp 2.307 Å; LiInd 2.320 Å). This finding represents the order of the electron density (+I effect of the substituents) within the C5-ring of the anions Cp*, Cp, and Ind. For completeness the nearly solvent-free structure of LiC_5Bz_5 in the compound $[(LiC_5Bz_5)_2C_6D_6]\cdot 2C_6D_6$ is mentioned here (one Li^+ ion is weakly bonded to a benzene ligand).[18] Two different Li^+ ions show interactions to the C5-rings with Li−C distances in the wide range of 2.11(2)−2.42(2) Å. Only one crystal structure of a Lewis base adduct of **1** is reported in the literature ([LiInd(tmeda)]; tmeda = tetramethylethylenediamine).[19] In this monomeric compound almost the same Li−C distances (2.330 Å) are observed as found in **1**.

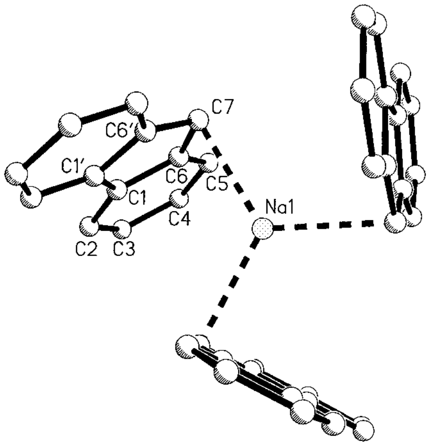

Figure 3. Coordination of three five-membered rings of the fluorenyl rings around the $Na(1)^+$ cation in NaFl (**2**). Distance (Å): Na(1)−C(7) 2.483(3). Angle (deg): C(7)−Na(1)-C(7′) 120°. Hydrogen atoms are not shown.

The crystallographic asymmetric unit of fluorenylsodium (**2**) in the space group $P6_3/m$ is formed by two independent sodium cations ($Na(1)^+$, site symmetry $\bar{6}$, and $Na(2)^+$, site symmetry 3) and half of the fluorenyl ring (mirror plane perpendicular to the fluorenyl ring; Figure 4). In the solid state, compound **2** can be described by the formula $Na_2[NaFl_3]$. Figure 3 shows the arrangement of $Na(1)^+$.

The sodium cation $Na(1)^+$ is coordinated by three C5-rings of three fluorenyl anions in a trigonal planar fashion forming the subunit $[NaFl_3]^{2-}$ (120° arrangement because of the 3-fold symmetry of the space group), which looks like a "propeller" and represents a tris(fluorenyl)sodate anion. We find η^1 bonding of the $Na(1)^+$ ion to the central carbon atom of the C5-ring (C(7)), with an extremely short Na−C distance of 2.483(3) Å. Two additional weak interactions are observed at the adjacent carbon atoms in the C5-ring (distances: Na(1)−C(6) 2.843(2) Å; Na(1)−C(6′) 2.843(2) Å). The distances of Na(1) to C(1) or the symmetry-related C(1′) are 3.343(3) Å and definitely out of range for bonding. The surroundings of the symmetry-related cations $Na(2)^+$ and $Na(2')^+$ are given in Figure 4.

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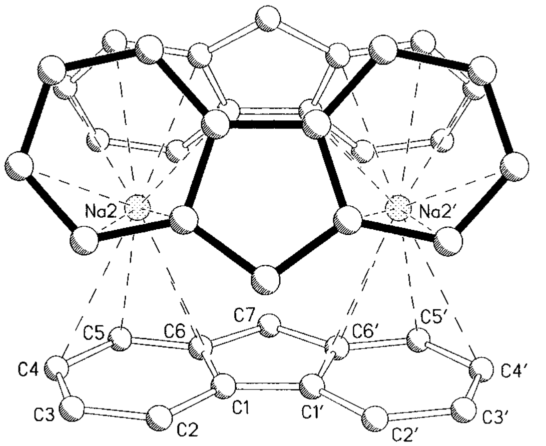

Figure 4. Surrounding of the "naked" sodium cation Na(2)$^+$ in NaFl (**2**). Distances (Å): Na(2)−C(1) 3.116(2); Na(2)−C(4) 3.055(2); Na(2)−C(5) 2.874(3); Na(2)−C(6) 2.906(2); Na(2)−Na(2′) 4.088(7); C···C 1.406(1) (equal restrained distances for all bonds within the fluorenyl ring).

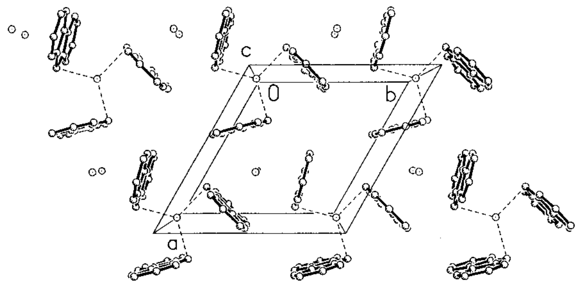

Figure 5. Drawing of the unit cell of NaFl (**2**). Hydrogen atoms are not shown.

The sodium cation Na(2)$^+$ can be described as a "naked" ion because it shows only weak Na−C interactions to three C6-rings of the fluorenyl anions. The following widely differing Na(2)−C distances are observed: two shorter distances (Na(2)−C(5) 2.874(3) Å and Na(2)−C(6) 2.906(2) Å), two medium distances (Na(2)−C(4) 3.055(2) Å and Na(2)−C(1) 3.116(2) Å), and two very long distances (Na(2)−C(3) 3.254(3) Å and Na(2)−C(2) 3.283(3) Å). This finding shows that the Na(2)$^+$ ion is not located perpendicularly above the centers of the C6-rings. Within the *ab* plane each Na(2)$^+$/Na(2′)$^+$ cation pair (Figure 4) is connected to three [Na(1)Fl$_3$]$^{2-}$ "propellers", which are mentioned above, building up a two-dimensional polymeric layer structure (Figure 5). Different layers are packed perpendicular to the crystallographic *c*-axis and do not interact with each other.

In the literature three base adducts of fluorenylsodium with nitrogen-containing ligands are reported: [NaFl(pmdta)], [NaFl(tmeda)]$_n$, and [NaFl(tmpda)]$_4$ (pmdta = pentamethyldiethylenetriamine, tmeda = tetramethylethylenediamine, tmpda = tetramethylpropylenediamine).[20] [NaFl(pmdta)] is a monomeric compound with a η^1 bonding of the Na$^+$ cation to the C5-ring (Na−C(η^1) distance 2.620(3) Å).[20] The second example, [NaFl(tmeda)]$_n$, crystallizes as a polymeric zigzag chain and shows the following similarity to the structure of **2**: The sodium cations are surrounded by three ligands, two fluorenyl anions and one neutral TMEDA ligand. Both fluorenyl anions are η^1 connected with the central carbon atom of the C5-ring to the Na$^+$ cations in the same manner as found for **2**. The distances here are significantly longer than found for **2** (Na−C(η^1) distances: [NaFl(tmeda)]$_n$, 2.634(3) Å (first fluorenyl ring), 2.773(3) Å (second fluorenyl ring); **2**, 2.483(3) Å). This makes perfect sense for two reasons: 1. The coordination number in [NaFl(tmeda)]$_n$ is 4, whereas it is 3 in **2**. 2. The two free pairs of electrons of the TMEDA ligand, which form dative bonds to the Na$^+$ cation, lower the positive charge at the Na$^+$ cation and cause a longer Na−C bonding. Also three ligands are connected to the sodium ions in the tetrameric complex [NaFl(tmpda)]$_4$ (third example) in a way very similar to that observed for [NaFl(tmeda)]$_n$.[20] After looking carefully at the structure of **2**, we now understand that the subunit [NaFl$_3$]$^{2-}$ has special stability because otherwise all Na$^+$ ions in **2** would be coordinated by the fluorenyl anions in a similar fashion, i.e., as a multidecker structure. It might be possible to separate this [NaFl$_3$]$^{2-}$ "propeller" with large stabilizing cations such as [PPh$_4$]$^+$, [S(NMe$_2$)$_3$]$^+$, or [Na(cryptand-221)]$^+$. These large cations are known as stabilizing cations, i.e., in the compounds [S(NMe$_2$)$_3$][MCp$_2$] (M = Li, Na, K),[22] [PPh$_4$][MCp$_2$][23,24] (M = Li, Na), and [Na(cryptand-221)][Fl].[25] It should be mentioned that the heavier p-block metals Sn and Pb show a similar so-called "paddle-wheel" type of structure which is realized in the complexes [Mg(thf)$_6$]$^+$-[MCp$_3$]$^-$ (M = Sn, Pb)[26] and that the nitrogen-containing carbazolyl anion (C$_8$H$_{12}$N$^-$) builds up a [Na(C$_8$H$_{12}$N)$_3$]$^{2-}$ propeller in the salt [Na(cryptand-221)]$^+{}_2$[Na(C$_8$H$_{12}$N)$_3$]$^{2-}$.[27]

The arrangement of the Na(2)$^+$ ions in **2** is related to the arrangement of the Li$^+$ ions in [LiFl]$_2$.[12] In both structures a pair of metal ions are coordinated by the C6-rings of the Fl$^-$ anions. Figure 4 shows three Fl$^-$ anions connected to this pair of Na$^+$ cations. In the compound [LiFl]$_2$, two Fl$^-$ anions are connected to the pair of Li$^+$ cations. It was surprising to find that the distance between the two Na$^+$ cations is 0.26 Å shorter than the distance between the two Li$^+$ cations (distances Na(2)−Na(2′) 4.088(7) Å and Li−Li′ 4.345(9) Å). To discover the reason behind this we had to consider the two opposite tendencies in this situation. The metal ions want to shift toward the C5-ring of the fluorenyl anion because the negative charge density is higher at the C5-ring (mostly at the central C atom of the C5-ring). The other tendency is that the positively charged metal cations cannot get too close together because of charge repulsion. This repulsion is for the Na(2)$^+$ ions a little bit smaller than for the Li$^+$ ions in [LiFl]$_2$ because the

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sodium ions are connected to three Fl$^-$ anions, which decreases the positive charge of the metal ions.

Experimental Section

General Procedures. All reactions were carried out by standard Schlenk techniques under purified argon. The solvents were heated under nitrogen over alkali metal (Na for toluene; K for *n*-hexane and THF; Na/K alloy for diethyl ether and cyclopentane) until a blue reaction of benzophenone occurred and then distilled. Indene was freshly distilled before use. The purity of the samples was checked by IR spectroscopy, NMR spectroscopy, and C, H analysis.

Synthesis of LiInd (1).[16] ^{n}BuLi (4.9 mL of a 1.6 M ^{n}BuLi/hexane solution; 7.8 mmol) was added to a solution of indene (1.00 g, 1.00 mL, 8.6 mmol) in diethyl ether (25 mL), yielding a red solution after stirring for 22 h at room temperature. After filtration, 20 mL of the diethyl ether were removed under reduced pressure, and cyclopentane (25 mL) was added. The resulting precipitate was isolated, washed with cyclopentane (twice, 10 mL), and dried at 75 °C and 10^{-3} Torr for 8 h. Yield: 0.67 g (70%) of a white microcrystalline powder. The compound is soluble in tetrahydrofuran and diethyl ether but not soluble in toluene or cyclopentane. Decomposition is above 90 °C. Anal. Calcd (found) for C_9H_7Li (**1**): C, 88.5 (87.8); H 5.8 (5.9). IR (ν, Nujol): 3089 (w), 3070 (w), 3051 (w), 3040 (w), 1335 (s), 1320 (vs), 1031 (s), 755 (vs), 731 (vs), 538 (m), 468 (s) cm^{-1}. ^{1}H NMR (200 MHz, THF-d_8): δ 7.28 (m, 2H, H$^{4/7}$), 6.51 (t, 1H, H^2), 6.41 (m, 2H, H$^{5/6}$), 5.89 (d, 2H, H$^{1/3}$) ppm. ^{13}C{^{1}H} NMR (50.3 MHz, THF-d_8): δ 130.9 (C$^{8/9}$), 120.7 (C$^{4/7}$), 116.6 (C^2), 115.0 (C$^{5/6}$), 92.7 (C$^{1/3}$) ppm.

Synthesis of NaFl (2).[28] Sodium hydride (0.21 g, 8.8 mmol, washed with *n*-hexane) and fluorene (1.58 g (9.5 mmol) were stirred in tetrahydrofuran for 22 h at 70 °C to yield a dark red solution, which was filtered and overlayered with *n*-hexane (33 mL). After 1 week big yellow crystals were obtained. The solution was decanted, and the remaining crystals were stirred with *n*-hexane for 12 h at room temperature. All crystals crumbled to a yellow precipitate of base-free fluorenylsodium. The precipitate was separated, washed with *n*-hexane (twice, 10 mL), and dried at 75 °C and 10^{-3} Torr for 8 h. Yield: 1.46 g (88%) of a microcrystalline yellow powder. The compound is soluble in tetrahydrofuran, dimethoxyethane, or diethyl ether, slightly soluble in toluene or benzene, but not soluble in *n*-hexane. Decomposition is above 90 °C. Anal. Calcd (found) for $C_{13}H_9Na$ (**2**): C, 83.0 (80.7); H 4.8 (5.3). IR (ν, Nujol): 3164 (vw), 3051 (m), 1322 (vs), 1223 (vs), 1195 (s), 985 (s), 773 (m), 754 (vs), 739 (s), 726 (vs) cm^{-1}. ^{1}H NMR (200 MHz, THF-d_8): δ 8.00 (d, 2H, H$^{2/2'}$), 7.41 (d, 2H, H$^{5/5'}$), 6.88 (t, 2H, H$^{4/4'}$), 6.52 (t, 2H, H$^{3/3'}$), 6.03 (s, 1H, H^7) ppm. ^{13}C{^{1}H} NMR (50.3 MHz, THF-d_8): δ 136.8 (C$^{6/6'}$), 127.2 (C$^{1/1'}$), 120.3 (C$^{4/4'}$), 119.5 (C$^{2/2'}$), 116.7 (C$^{5/5'}$), 109.9 (C$^{3/3'}$), 80.4 (C^7) ppm.

X-ray Crystallography. X-ray powder diffraction methods were used to determine the solid-state structures of the compounds. All crystallographic results are given in Table 1 and Figures 1−5. The intensity data were collected with high-resolution synchrotron radiation in transmission geometry at room temperature (beamline X3B1, National Synchrotron Light Source, Brookhaven National Laboratory). For this purpose the moisture- and air-sensitive compounds were sealed in 0.7 mm glass capillaries under argon. The powder patterns were indexed with the program ITO.[29] We used the program SIRPOW92 and direct methods in order to determine the positions of the metal and carbon atoms.[30] All C···C bond lengths within the indenyl or fluorenyl rings were restrained so they would have the same value (rigid body refinement). The positions of the hydrogen atoms were calculated with a C−H distance of 1.05 Å. For the sample of LiInd, we could not find any nonfitting peaks from other crystalline phases or amorphous background in the Rietveld refinement plots. For the sample NaFl, traces of excessive fluorene (<4%) were visible in the powder pattern. Therefore fluorene was included in the refinement process as a second phase, using the data from a single crystal study as the starting parameters.[31] Final Rietveld refinements were carried out with the program package GSAS (see figures in Supporting Information).[32]

Table 1. Crystallographic Data for LiInd (1) and NaFl (2)

	1	2
formula	C_9H_7Li	$C_{13}H_9Na$
fw	122.10	188.20
cryst syst	orthorhombic	hexagonal
space group	*Pnma* (No. 62)	$P6_3/m$ (No. 176)
a (Å)	20.5001(1)	9.2828(1)
b (Å)	3.9645(1)	
c (Å)	8.3086(1)	19.1613(2)
V (Å^3)	675.26(1)	1429.93(2)
Z	4	6
d_{calcd} (g cm^{-3})	1.201	1.311
2θ range (deg)	3.0−59.9	2.0−50.1
$\delta(2\theta)$ (deg; s)[a]	0.004; 5.2	0.005; 4.3
λ (Å)	1.148 94(1)	1.149 66(2)
no. of reflcns[b]	281	212
no. of params	23	20
R_{wp}	0.069	0.055
R_p	0.048	0.043
R_{F2}	0.072	0.080

[a] Scan steps with seconds for each step. [b] From Le-Bail fit.

Acknowledgment. The authors are grateful to Prof. Dr. P. W. Stephens for his help during data collection. Research was carried out, in part, at the National Synchrotron Light Source at Brookhaven National Laboratory, which is supported by the U.S. Department of Energy, Division of Materials Sciences, and Division of Chemical Sciences. The SUNY X3 beamline at NSLS is supported by the Division of Basic Energy Sciences of the U.S. Department of Energy under Grant DE-FG02-86ER45231. Support by the Deutsche Forschungsgemeinschaft (Be 760/10-1 and Di 687/2-1) is gratefully acknowledged. F.O. wishes to thank Sachsen-Anhalt for a Habilitation fellowship.

Supporting Information Available: Text giving crystallographic details, tables of all bond lengths and angles, atomic coordinates and equivalent thermal parameters, and ORTEP diagrams Rietveld plots of indenyllithium and fluorenylsodium (13 pages). This material is available free of charge via the Internet at http://pubs.acs.org.

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