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# The formation and molecular structure of $[Na(THF)_6][(\eta^5\text{-}C_9H_7)_3Pr(\mu\text{-}Cl)Pr(\eta^5\text{-}C_9H_7)_3]$

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**Abstract**

The reaction of 1 : 0.8 mole ratio of anhydrous $PrCl_3$ with sodium indenyl in tetrahydrofuran (THF) results in the formation of $[Na(THF)_6][(\eta^5\text{-}C_9H_7)_3Pr(\mu\text{-}Cl)Pr(\eta^5\text{-}C_9H_7)_3]$ with a good yield. X-ray analysis demonstrates that the complex consists of a disconnected ion pair formed by $[(\eta^5\text{-}C_9H_7)_3Pr(\mu\text{-}Cl)Pr(\eta^5\text{-}C_9H_7)_3]$ anion and the sodium cation surrounded by six THF molecules. The complex crystallizes in triclinic system, of space group $P1$ with unit-cell constants $a = 10.693(2)$, $b = 12.687(2)$, $c = 12.825(2)$ Å, $\alpha = 92.48(2)$, $\beta = 82.17(2)$, $\gamma = 91.73(1)°$, $D_c = 1.41$ g cm$^{-3}$, $V = 1721.7(5)$ Å$^3$ for $Z = 1$. The structure was solved by direct methods and full-matrix least-squares refinement to the final $R = 0.062$, $R_w = 0.072$.

*Keywords:* Praseodymium; Indenyl; Molecular structure

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## 1. Introduction

In the past few decades, the chemistry of organolanthanide complexes has become one of the most active field of organometallic chemistry [1–4]. With respect to lanthanide–indenyl complexes, the synthesis and characterization of $(C_9H_7)_3Ln \cdot THF$ (THF≡tetrahydrofuran) were reported by Tsutsui and Gysling [5] and Huang et al. [6] respectively, $(C_9H_7)_2LnCl \cdot THF$ and $(C_9H_7)LnCl_2 \cdot THF$ by Chen and coworkers [7,8], and some other indenyl-containing complexes, such as $(C_9H_7)_3Sm$ [9], $(C_9H_7)GdCl_2 \cdot 3THF$ [10], $(C_9H_7)Pr(C_8H_8) \cdot 2THF$ [11], and $[Na(THF)_6][(\eta^5\text{-}C_9H_7)_3Ln(\mu\text{-}Cl)Ln(\eta^5\text{-}C_9H_7)_3]$ (Ln≡Nd [12] or Sm [13]), have been synthesized and characterized by X-ray diffraction analysis etc. In an attempt to prepare $(C_9H_7)PrCl_2 \cdot 3THF$ as an intermediate, a fine crystal of $[Na(THF)_6][(\eta^5\text{-}C_9H_7)_3Pr(\mu\text{-}Cl)Pr(\eta^5\text{-}C_9H_7)_3]$, instead of $(C_9H_7)PrCl_2 \cdot 3THF$, was obtained as a result. To the best of our knowledge, the synthesis and crystal structure of $[Na(THF)_6][(\eta^5\text{-}C_9H_7)_3Pr(\mu\text{-}Cl)Pr(\eta^5\text{-}C_9H_7)_3]$ have not been reported. So, we decided to carry out a structural analysis of the title complex.

## 2. Results and discussion

The complex of $[Na(THF)_6][(\eta^5\text{-}C_9H_7)_3Pr(\mu\text{-}Cl)Pr(\eta^5\text{-}C_9H_7)_3]$ crystallizes in the triclinic system, with space group $P1$ and unit-cell constants $a = 10.693(2)$, $b = 12.687(2)$, $c = 12.825(2)$ Å, $\alpha = 92.48(2)$, $\beta = 82.17(2)$, $\gamma = 91.73(1)°$, $D_c = 1.41$ g cm$^{-3}$, $V = 1721.7(5)$ Å$^3$ for $Z = 1$. The final positional and thermal parameters are listed in Table 1; selected bond lengths and bond angles are listed in Table 2 and Table 3 respectively. Fig. 1 and Fig. 2 give the perspective view of the complex structure and the package of the molecule in unit cell respectively.

In several of our previous studies [14–20], we found that organolanthanide complexes with both Cp and other ligands are comparatively unstable upon heating; disproportionation would take place, owing to the arrangement of ligands. We also found that complexes of the type $CpLnL_2$ (L = ligand) are less thermally stable than those of $Cp_2LnL$, and the final disproportionation products of both types of complex are $Cp_3Ln$ and

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Table 1
Positional and thermal parameters

| Atom | *x* | *y* | *z* | $B_{eq}$ (Å) |
|---|---|---|---|---|
| Pr | 0.87421(4) | 0.15241(3) | 0.90105(3) | 3.69(2) |
| Cl | 1.0000 | 0 | 1.0000 | 5.4(1) |
| Na | $\frac{1}{2}$ | $\frac{1}{2}$ | $\frac{1}{2}$ | 4.5(2) |
| O(1) | 0.4372(7) | 0.4861(7) | 0.3281(5) | 8.1(4) |
| O(2) | 0.4262(7) | 0.3216(5) | 0.5242(5) | 7.1(4) |
| O(3) | 0.7085(6) | 0.4344(6) | 0.4366(6) | 7.6(4) |
| C(11) | 1.0836(7) | 0.3009(6) | 0.8165(6) | 4.2(3) |
| C(12) | 0.9872(8) | 0.2976(7) | 0.7491(6) | 5.0(4) |
| C(13) | 0.9859(8) | 0.1984(8) | 0.7013(6) | 5.6(4) |
| C(14) | 1.0688(8) | 0.1329(6) | 0.7401(7) | 5.3(4) |
| C(15) | 1.1333(7) | 0.1964(5) | 0.8121(5) | 4.1(3) |
| C(16) | 1.2325(8) | 0.1775(6) | 0.8719(6) | 4.9(4) |
| C(17) | 1.2812(8) | 0.2594(8) | 0.9267(7) | 5.6(5) |
| C(18) | 1.2336(8) | 0.3611(7) | 0.9275(6) | 5.4(4) |
| C(19) | 1.1364(8) | 0.3822(6) | 0.8752(6) | 4.8(4) |
| C(21) | 0.6987(7) | 0.2352(6) | 1.0819(6) | 4.6(4) |
| C(22) | 0.7233(9) | 0.3142(7) | 1.0051(7) | 5.9(5) |
| C(23) | 0.8479(9) | 0.3483(6) | 1.0007(7) | 5.6(5) |
| C(24) | 0.9086(7) | 0.2883(6) | 1.0678(6) | 4.7(4) |
| C(25) | 0.8156(7) | 0.2198(6) | 1.1210(6) | 4.2(3) |
| C(26) | 0.8195(7) | 0.1474(7) | 1.2005(6) | 4.8(4) |
| C(27) | 0.7099(8) | 0.0969(6) | 1.2410(7) | 5.3(4) |
| C(28) | 0.5959(8) | 0.1130(7) | 1.2039(7) | 5.6(4) |
| C(29) | 0.5892(7) | 0.1809(7) | 1.1261(7) | 5.6(4) |
| C(31) | 0.7380(1) | 0.0420(1) | 0.7432(8) | 8.0(7) |
| C(32) | 0.6720(2) | 0.1210(1) | 0.7730(3) | 20.0(2) |
| C(33) | 0.6230(1) | 0.1010(2) | 0.8770(1) | 12.0(1) |
| C(34) | 0.6705(9) | 0.0162(9) | 0.9153(8) | 6.1(5) |
| C(35) | 0.7500(1) | −0.0352(9) | 0.8270(1) | 9.0(8) |
| C(36) | 0.8000(1) | −0.1260(1) | 0.8180(1) | 9.5(9) |
| C(37) | 0.8630(1) | −0.1620(1) | 0.7170(2) | 11.0(1) |
| C(38) | 0.8650(1) | −0.0890(2) | 0.6370(1) | 10.0(1) |
| C(39) | 0.8170(2) | −0.0050(2) | 0.6220(2) | 15.0(1) |
| C(41) | 0.307(1) | 0.472(2) | 0.309(1) | 15.0(1) |
| C(42) | 0.301(2) | 0.452(2) | 0.207(1) | 14.0(1) |
| C(43) | 0.431(2) | 0.459(2) | 0.152(1) | 12.0(1) |
| C(44) | 0.510(1) | 0.471(1) | 0.232(1) | 10.0(1) |
| C(51) | 0.433(2) | 0.245(1) | 0.439(1) | 10.0(1) |
| C(52) | 0.457(1) | 0.1431(9) | 0.485(1) | 8.7(7) |
| C(53) | 0.391(1) | 0.1546(8) | 0.595(1) | 8.3(7) |
| C(54) | 0.384(2) | 0.268(1) | 0.614(1) | 12.0(1) |
| C(61) | 0.749(2) | 0.336(1) | 0.459(1) | 12.0(1) |
| C(62) | 0.885(1) | 0.338(1) | 0.442(1) | 9.3(8) |
| C(63) | 0.915(1) | 0.418(1) | 0.360(1) | 9.5(8) |
| C(64) | 0.803(1) | 0.476(2) | 0.368(2) | 16.0(1) |

$LnL_3$. Recently, we [21] reported that the reaction of $CpSmCl_2 \cdot 3THF$ with sodium allyoxypropynylide ($NaC{\equiv}CCH_2OCH_2CH{=}CH_2$) in a mole ratio of 1 : 2 resulted in the formation of disproportionation product of $Cp_3SmTHF$. Moreover, Rogers et al. [22] have also pointed out that the formation of $Cp_3Gd \cdot THF$ is the result of the disproportionation reaction of $CpGdCl_2$ in THF solution. The same situation has been encountered in the reaction of anhydrous $PrCl_3$ and sodium indenyl in THF in a 1 : 0.8 mole ratio with the formation of $[Na(THF)_6][(\eta^5\text{-}C_9H_7)_3Pr(\mu\text{-}Cl)Pr(\eta^5\text{-}C_9H_7)_3]$ as the only pure product. So, we conclude that the formation of the title complex is the result of disproportionation reaction and the formation pathway is suggested to be as follows:

$$6(C_9H_7)Na + 6PrCl_3 \longrightarrow 6[(C_9H_7)PrCl_2 \cdot nTHF] + 6NaCl$$

$$\Big\downarrow \text{disproportionation}$$

$$2(C_9H_7)_3Pr + 4PrCl_3$$

$$\Big\downarrow \text{NaCl.6THF}$$

$$[Na(THF)_6][\eta^5\text{-}C_9H_7)_3Pr(\mu\text{-}Cl)Pr(\eta^5\text{-}C_9H_7)_3]$$

The complex is sensitive to air and moisture; it will decompose in a matter of minutes upon exposure to air; it is soluble in THF and insoluble in toluene or *n*-hexane. An IR spectrum study shows characteristic absorptions of indenyl group at 3062, 1612, 1470, 1019 and 765 $cm^{-1}$, that of Pr–Cl at 446 $cm^{-1}$, and those of THF at 947 and 1047 $Cm^{-1}$.

From Fig. 1, we can see that the title complex consists of a disconnected ion pair formed by the $[(\eta^5\text{-}C_9H_7)_3Pr(\mu\text{-}Cl)Pr(\eta^5\text{-}C_9H_7)_3]$ anion and the sodium cation surrounded by six THF molecules. In the anion each praseodymium atom is surrounded by three indenyl ligands and chloride which connects the two praseodymium atoms. All carbon atoms in each of indenyl ligands are coplanar with mean deviations of 0.0364, 0.0269 and 0.0403 Å respectively, and the anion itself has crystallographic $C_i$ symmetry for the planes of Ind(1) and Ind(1′); the planes of Ind(2) and Ind(2′), and the planes of Ind(3) and Ind(3′) are parallel, respectively, and the chloride is situated at the crystallographic symmetrical centre. The dihedral angles of the plane of Ind(1) and Ind(2), of the plane of Ind(2) and Ind(3) and of the plane of Ind(3) and Ind(1) are

Table 2
The selected bond lengths (Å)

| | | | |
|---|---|---|---|
| Pr–Cl | 2.8272(6) | Pr–C(11) | 3.000(7) |
| Pr–C(12) | 2.862(7) | Pr–C(13) | 2.754(8) |
| Pr–C(14) | 2.732(8) | Pr–C(15) | 2.897(7) |
| Pr–C(21) | 2.954(7) | Pr–C(22) | 2.824(8) |
| Pr–C(23) | 2.749(8) | Pr–C(24) | 2.745(7) |
| Pr–C(25) | 2.902(7) | Pr–C(31) | 2.937(7) |
| Pr–C(32) | 2.900(2) | Pr–C(33) | 2.800(1) |
| Pr–C(34) | 2.730(9) | Pr–C(35) | 2.876(9) |
| C(11)–C(12) | 1.43(1) | C(12)–C(13) | 1.38(1) |
| C(13)–C(14) | 1.39(1) | C(14)–C(15) | 1.43(1) |
| C(15)–C(11) | 1.44(1) | C(21)–C(22) | 1.43(1) |
| C(22)–C(23) | 1.43(1) | C(23)–C(24) | 1.41(1) |
| C(24)–C(25) | 1.42(1) | C(25)–C(21) | 1.43(1) |
| C(31)–C(32) | 1.26(1) | C(32)–C(33) | 1.40(1) |
| C(33)–C(34) | 1.35(1) | C(34)–C(35) | 1.46(1) |
| C(35)–C(31) | 1.50(1) | | |
| Na–O(1) | 2.388(6) | Na–O(1b) | 2.388(6) |
| Na–O(2) | 2.393(6) | Na–O(2b) | 2.393(6) |
| Na–O(3) | 2.426(6) | Na–O(3b) | 2.426(6) |

Table 3
Selected bond angles

| | | | |
|---|---|---|---|
| C(14)–Pr–C(13) | 29.3(3) | C(14)–Pr–C(15) | 29.3(2) |
| C(12)–Pr–C(13) | 28.3(2) | C(14)–Pr–C(12) | 48.0(3) |
| C(12)–Pr–C(15) | 47.1(2) | C(13)–Pr–C(15) | 47.1(2) |
| C(24)–Pr–C(23) | 29.7(3) | C(23)–Pr–C(22) | 28.7(3) |
| C(24)–Pr–C(25) | 29.0(2) | C(22)–Pr–C(25) | 47.1(2) |
| C(24)–Pr–C(22) | 48.2(3) | C(23)–Pr–C(25) | 47.5(2) |
| C(34)–Pr–C(33) | 28.2(4) | C(34)–Pr–C(35) | 30.1(3) |
| C(33)–Pr–C(32) | 28.3(7) | C(35)–Pr–C(32) | 47.8(4) |
| C(34)–Pr–C(32) | 48.2(6) | C(33)–Pr–C(35) | 46.8(5) |
| Cl–Pr–C(12) | 126.6(2) | Cl–Pr–C(13) | 114.6(2) |
| Cl–Pr–C(14) | 85.5(2) | Cl–Pr–C(15) | 79.6(2) |
| Cl–Pr–C(22) | 125.7(2) | Cl–Pr–C(23) | 116.9(2) |
| Cl–Pr–C(24) | 87.3(2) | Cl–Pr–C(25) | 78.7(2) |
| Cl–Pr–C(32) | 128.7(3) | Cl–Pr–C(14) | 115.5(4) |
| Cl–Pr–C(34) | 87.4(3) | Cl–Pr–C(35) | 80.9(3) |
| Pr–Cl–Pr(a) | 180(2) | O(1)–Na–O(1b) | 180(5) |
| O(2)–Na–O(2b) | 180.00 | O(3)–Na–O(3b) | 180(5) |
| O(1)–Na–O(2) | 88.1(3) | O(1)–Na–O(2b) | 91.9(3) |
| O(1)–Na–O(3) | 92.4(3) | O(1)–Na–O(3b) | 87.6(3) |
| O(2)–Na–O(3) | 88.8(2) | O(2)–Na–O(3b) | 91.2(2) |
| C(14)–C(15)–C(11) | 107.6(7) | C(12)–C(11)–C(15) | 106.6(7) |
| C(13)–C(12)–C(11) | 107.7(7) | C(12)–C(13)–C(14) | 111.2(7) |
| C(13)–C(14)–C(15) | 106.8(7) | C(22)–C(21)–C(25) | 106.4(7) |
| C(23)–C(22)–C(21) | 108.8(8) | C(22)–C(23)–C(24) | 109.3(7) |
| C(23)–C(24)–C(25) | 107.3(7) | C(24)–C(25)–C(21) | 108.1(7) |
| C(32)–C(31)–C(35) | 116.0(2) | C(31)–C(32)–C(33) | 104.0(2) |
| C(34)–C(33)–C(32) | 114.0(2) | C(33)–C(34)–C(35) | 106.0(1) |
| C(34)–C(35)–C(31) | 99.0(1) | | |

63.47, 112.00 and 64.85° respectively. The centriods of each five-membered ring in indenyl groups and chloride around each praseodymium atom form a distorted tetrahedron, and the chloride is the one conjugate apex of the two tetrahedra. The sodium cation is coordinated by six THF molecules and has a slightly distorted octahedron coordination geometry with oxygen atoms of the THF molecules as apices. So, the crystal structure of the title complex is very similar to those of the reported complexes of [Na(THF)$_6$][($\eta^5$-C$_9$H$_7$)$_3$Ln($\mu$-Cl)Ln($\eta^5$-C$_9$H$_7$)$_3$] (Ln≡Nd [12] or Sm [13]). In these complexes, the coordination number of each lanthanide atom is ten.

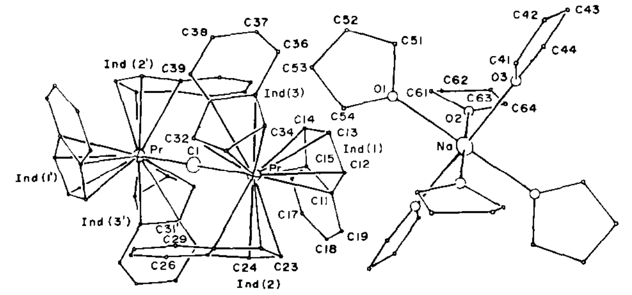

Fig. 1. The molecular structure of [Na(THF)$_6$][($\eta^5$-C$_9$H$_7$)$_3$Pr($\mu$-Cl)Pr($\eta^5$-C$_9$H$_7$)$_3$].

As the two complexes of [Na(THF)$_6$][($\eta^5$-C$_9$H$_7$)$_3$-Ln($\mu$-Cl)Ln($\eta^5$-C$_9$H$_7$)$_3$] (Ln≡Nd [12] or Sm [13]) and the title complex [Na(THF)$_6$][($\eta^5$-C$_9$H$_7$)$_3$Pr($\mu$-Cl)Pr-($\eta^5$-C$_9$H$_7$)$_3$] all crystallize in triclinic system and belong to the same space group *P*1, so, these three complexes are isomorphous. It is natural to make a comparison among the bond lengths of Ln–Cl in the series of [Na(THF)$_6$][($\eta^5$-C$_9$H$_7$)$_3$Ln($\mu$-Cl)Ln($\eta^5$-C$_9$H$_7$)$_3$] (Ln≡Pr, Nd or Sm), the bond lengths of Ln–Cl are in the sequence of Pr–Cl (2.827 Å) < Nd–Cl < (2.841 Å) < Sm–Cl (2.899 Å), which is opposite to the decreasing trend of lanthanide ionic radii [23]. We suppose that these results are presumably due to mutual interaction of many factors: the crowded degree of indenyl ligands around each lanthanide ion, the repulsion forces of indenyl ligands presented separately around two lanthanide ions and the ionic radii of lanthanides.

In the cation, the bond lengths of Na–0 range from 2.388 to 2.426 Å, an average of 2.402 Å which is similar to those of the reported neodymium and samarium complexes.

## 3. Experimental details

All manipulations were carried out in an atmosphere of purified argon using Schlenk techniques; all solvents were refluxed and distilled over the sodium ketyl of benzophenone under argon immediately before use. Elemental analysis data were obtained with a Yanaco MT-2 analyser; the analysis for Pr was obtained by the published method [24]. IR spectra were recorded on Perkin–Elmer 983(G) spectrometer; the melting point was determined in sealed argon-filled capillaries without correction. Sodium indenyl was prepared by reaction of sodium hydride and indene in

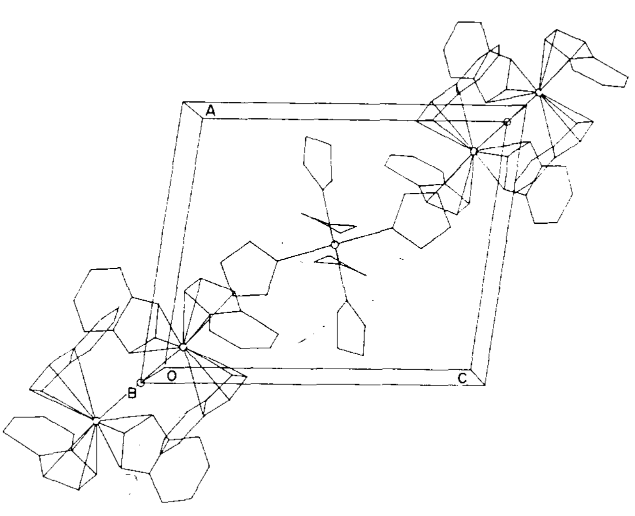

Fig. 2. The package of the molecule inside the cell.

THF. Anhydrous $PrCl_3$ was prepared according to the published procedure [25].

### 3.1. *The formation of* $[Na(THF)_6][(\eta^5\text{-}C_9H_7)_3Pr(\mu\text{-}Cl)Pr(\eta^5\text{-}C_9H_7)_3]$

To the suspension of 2.894 g (11.70 mmol) of anhydrous $PrCl_3$ in THF was added 15 ml (9.36 mmol) THF solution of sodium indenyl; the reaction was stirred at room temperature overnight, after the usual centrifugation. Yellow–green crystals (1.75 g; yield, 30.6%; based on $PrCl_3$) were obtained upon cooling the clear solution at −20°C for several days. Anal. Found, 20.21; C, 63.52; H; 6.34 Calc.: Pr, 19.25; C, 63.94; H, 6.15. IR: a 3062, 2919, 1612, 1377, 1019, 947, 765, 690, 446 $cm^{-1}$. Melting point, 84°C.

### 3.2. *Crystallography of* $[Na(THF)_6][(\eta^5\text{-}C_9H_7)_3Pr(\mu\text{-}Cl)Pr(\eta^5\text{-}C_9H_7)_3]$

A single crystal of the title complex with dimensions $0.58 \times 0.42 \times 0.28$ mm was selected and sealed in a thin-walled glass capillary under argon and used in the data collections. The intensity data and unit-cell parameters were collected on an Enraf–Nonius CAD4 diffractometer with Mo Kα (graphite monochromator; $\lambda = 0.71069$ Å) in the range $3° < 2\theta < 50°$ by $2\theta\text{–}\omega$ scanning. Corrections for Lorentz, polarization and absorption with the empirical scanning technique were applied, 6387 reflections were measured; 5470 reflections with $I > 3\sigma(I)$ of the 6029 independent reflections were considered observed and used in the structure determination.

The praseodymium atoms and chloride were located by direct methods; the positions of all non-hydrogen atoms were revealed by successive Fourier synthesis. Hydrogen atoms were introduced in calculated positions. All positional and thermal parameters for non-hydrogen atoms were refined anisotropically with full-matrix least-squares methods and converged to final $R = 0.062$, $R_w = 0.072$ ($w = 1/\sigma^2(F_i)$) and $(\Delta/\sigma)_{max} = 0.24$; the final difference Fourier map of the complex showed no peak higher than 1.66 electrons Å$^{-3}$ or less than −1.28 electrons Å$^{-3}$. All calculation were performed on a Micro Vax II computer with the TEXSAN v2.1 package.

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