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Heterobimetallic Heterocyclic Derivatives of Indium(III)

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ABSTRACT: *Reactions of $InCl_3$ with potassium salts of bifunctional tridentate (L^1H_2=HOC_6H_4CH=$NCH_2CHMeOH$) and monofunctional bidentate (L^2H=HOC_6H_4CH=N-i-Bu) Schiff bases in 1:1 and 1:2 molar ratio in benzene afford complexes $In(L^1)Cl$ and $In(L^2)_2Cl$, respectively. On reaction with potassium isopropoxymetallates $KB(O\text{-}i\text{-}Pr)_4$, $KAl(O\text{-}i\text{-}Pr)_4$, $KTi(O\text{-}i\text{-}Pr)_5$, and $KNb(O\text{-}i\text{-}Pr)_6$, they produce interesting heterobimetallic heterocyclic complexes. These are characterized by elemental (N, B, Al, Ti, and Nb) analyses, molecular weight measurements, and spectral [IR, NMR (^1H, ^{13}C, ^{11}B, and ^{27}Al)] studies. Probable structures are suggested for them.* © 2003 Wiley Periodicals, Inc. Heteroatom Chem 15:21–25, 2004; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.10206

INTRODUCTION

Although there have been some reports on complexes of indium(III) derived from monofunctional bidentate and bifunctional tri- and tetra-dentate Schiff bases [1], heterobimetallic heterocyclic derivatives of indium supported by these types of ligands are not known. It is also worthy to note that only one report on heterometallic isopropoxide, $In[Al(O\text{-}i\text{-}Pr)_4]_3$ [2], is available in the literature.

In view of the above, it was considered worthwhile to investigate the heterobimetallic chemistry of indium(III) complexes derived from bifunctional tridentate and monofunctional bidentate Schiff bases. The studies on heterometallic systems presented herein assume special significance in view of the presence of NMR active nuclei such as ^{11}B and ^{27}Al in the molecular species, which would help considerably in elucidating coordination environment around these atoms [3,4].

In this paper, we report synthesis and characterization of heterobimetallic heterocyclic complexes of indium derived from monofunctional bidentate and bifunctional tridentate Schiff bases.

RESULTS AND DISCUSSION

Reactions of anhydrous $InCl_3$ with potassium salts of $HOC_6H_4CH{=}NCH_2CHMeOH$ (L^1H_2) and $HOC_6H_4CH{=}N\text{-}i\text{-}Bu$ (L^2H) in 1:1 and 1:2 molar ratio respectively, in benzene, afford the derivatives **1** and **2**.

$$InCl_3 + K_2L^1 \xrightarrow[\text{reflux}]{\text{benzene}} \underset{\mathbf{1}}{In(L^1)Cl} + 2KCl\downarrow$$

$$InCl_3 + 2KL^2 \xrightarrow[\text{reflux}]{\text{benzene}} \underset{\mathbf{2}}{In(L^2)_2Cl} + 2KCl\downarrow$$

With $KB(O\text{-}i\text{-}Pr)_4$, $KAl(O\text{-}i\text{-}Pr)_4$, $KTi(O\text{-}i\text{-}Pr)_5$, and $KNb(O\text{-}i\text{-}Pr)_6$, **1** and **2** afford heteronuclear derivatives **3** and **4**, respectively.

$$\mathbf{1} + K[M(O\text{-}i\text{-}Pr)_n] \xrightarrow[\text{reflux}]{\text{benzene}} \underset{\mathbf{3}}{In(L^1)\{M(O\text{-}i\text{-}Pr)_n\}} + KCl\downarrow$$

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3a: M = B, $n = 4$; **3b**: M = Al, $n = 4$; **3c**: M = Ti, $n = 5$; **3d**: M = Nb, $n = 6$.

$$2 + K[M(O\text{-}i\text{-}Pr)_n] \xrightarrow[\text{reflux}]{\text{benzene}} \underset{\mathbf{4}}{In(L^2)_2\{M(O\text{-}i\text{-}Pr)_n\}} + KCl\downarrow$$

4a: M = B, $n = 4$; **4b**: M = Al, $n = 4$; **4c**: M = Ti, $n = 5$; **4d**: M = Nb, $n = 6$.

The new heteronuclear derivatives **3** and **4** (Table 1) are moisture-sensitive, yellow solids or viscous liquids, soluble in common organic solvents (e.g., benzene, chloroform, etc.); and they also exhibit monomeric behaviour in benzene solution.

IR Data

The IR spectra of indium compounds **1–4** (Table 2) show a medium intense band at 1150–1210 cm^{-1} due to ν(C—O) (phenolate). A sharp band due to ν(C=N) has been observed at 1610 cm^{-1} in **1** and **2** derivatives. In heterometallic derivatives **3** and **4**, ν(C=N) appeared at 1637.5. The shift to higher wave number is indicative of the weaker coordination of azomethine nitrogen in heterobimetallic derivatives. The additional weak band at 450–475 cm^{-1} may be assigned to ν(In ← N). A medium intensity band observed at 550–580 cm^{-1} has been assigned to ν(In—O) [5,6]. In heteronuclear derivatives **3** and **4**, additional bands due to ν(M—O) are observed at 1280–1300 (M = B), 750–662.5 (M = Al), 570 (M = Ti), and 560 (M = Nb) cm^{-1}.

NMR Data

The singlet for the azomethine proton (Table 3) observed at δ 8.02 in **1** and **2** exhibits a small (0.28 ppm) downfield shift compared to its position in the precursor derivative.

The spectra of heterobimetallic derivatives **3** and **4** exhibit signals due to the ligand moiety along with two sets of signals (doublets and septets) for terminal and bridging isopropoxy groups in the region δ 3.29–3.67, 3.77–4.18 {C**H**(CH$_3$)$_2$} and δ 0.53–0.72, 0.82–0.98 {CH(C**H**$_3$)$_2$}.

In the ^{13}C NMR spectrum of **1** (Table 4), signals for >**C**=N, (CH$_3$)C**H**O, and **C**—O groups appear at δ 167.6, 62.7, and 170.8, respectively. A downfield shift has been observed in the positions of these signals as compared to the positions in the parent ligand. These observations are indicative of the involvement of the hetero atoms of >C=**N**, (CH$_3$)CH**O**, and C—**O** groups in bonding. The signals for alkyl carbon do not show any appreciable shift with respect to their positions in the ligand.

^{13}C NMR spectra of **3** show a downfield shift in the positions of signals for the carbons **C**=N,

TABLE 1 Analytical and Physical Properties of **1–4**

						Analysis (%) Found (Calcd.)		
	Empirical Formula	*State*	*Yield (%)*	*m.p. (°C)*	*g KCl Found (Calcd.)*	*In*	*N*	*Cl/B/Al/Ti/Nb*
1	$C_{10}H_{11}ClInNO_2$	Yellow solid	88	280 (decom.)	0.79 (0.80)	34.24 (35.06)	4.10 (4.27)	9.8 (10.82)
2	$C_{22}H_{28}ClInN_2O_2$	Yellow solid	99	220–230	0.41 (0.41)	24.92 (25.70)	6.18 (6.27)	7.86 (7.94)
3a	$C_{22}H_{39}BInNO_6$	Yellow solid	92	290 (decom.)	0.12 (0.12)	20.1 (21.29)	2.46 (2.59)	2.00 (2.00)
3b	$C_{22}H_{39}AlInNO_6$	Yellow solid	97	220 (decom.)	0.20 (0.21)	19.98 (20.67)	2.48 (2.52)	4.68 (4.86)
3c	$C_{25}H_{46}InNO_7Ti$	Yellow solid	90	270 (decom.)	0.12 (0.12)	18.00 (18.07)	2.20 (2.20)	7.42 (7.53)
3d	$C_{28}H_{53}InNbNO_8$	Yellow viscous liq.	99	–	0.09 (0.09)	15.49 (15.52)	1.78 (1.89)	12.48 (12.56)
4a	$C_{34}H_{56}BInN_2O_6$	Yellow viscous liq.	93	–	0.11 (0.12)	16.98 (17.43)	4.2 (4.25)	1.58 (1.64)
4b	$C_{34}H_{56}AlInN_2O_6$	Yellow solid	93	260 (decom.)	0.12 (0.14)	17.00 (17.02)	4.09 (4.15)	3.92 (4.00)
4c	$C_{37}H_{63}InN_2O_7Ti$	Yellow solid	92	265 (decom.)	0.12 (0.12)	15.00 (15.21)	3.58 (3.71)	6.12 (6.34)
4d	$C_{40}H_{70}InNbN_2O_8$	Yellow solid	99	268 (decom.)	0.09 (0.09)	13.20 (13.37)	3.12 (3.26)	10.62 (10.81)

TABLE 2 IR Data (cm^{-1}) of **1–4**

	ν(C=N)	ν(C–O)	ν(In–O)	ν(In ← N)	ν(In–Cl) ν(M–O)
1	1610	1150	600	450	250
2	1610	1150	600	450	250
3a	1637.5	1150	600	450	1350
3b	1637.5	1150	600	475	762.5
3c	1637.5	1210	600	462.5	570
3d	1637.5	1210	600	412.5	580
4a	1650	1150	600	450	1350
4b	1637.5	1150	600	450	750
4c	1637.5	1210	600	462.5	570
4d	1637.5	1210	600	462.5	580

$(CH_3)H\mathbf{C}O$, and **C**–O groups, when compared with the analogous signals in **1**. Two sets of signals due to bridging and terminal isopropoxy groups have been observed in the spectra of all the new heterobimetallic derivatives at δ 66.23 $\mathbf{C}H(CH_3)_2$, 29.61 $CH(\mathbf{C}H_3)_2$ and δ 65.29 $\mathbf{C}H(CH_3)_2$, 25.24 $(CH(\mathbf{C}H_3)_2)$, respectively.

3b and **4b** exhibit ^{27}Al signals at δ 43.0 and 38.15 indicating the tetracoordinate environment [4] around the aluminium atom.

^{11}B signals at δ 1.8 and 3.39 for **3a** and **4a** support the presence of tetracoordinated boron [3]. In view of the above spectroscopic findings plausible structures are shown in Figs. 1–3.

The titanium and niobium analogs, with three and four terminal isopropoxy groups attached to them, adopt pentagonal bipyramidal and octahedral geometry, respectively.

EXPERIMENTAL

All reactions and subsequent manipulations were performed under rigorously moisture free environment. Benzene, toluene, and *n*-hexane (BDH) were dried by refluxing over sodium benzophenone ketyl. Ethyl alcohol (b.p. 78°C) was kept over freshly ignited calcium oxide and distilled. The distillate was then dried by refluxing over magnesium ethoxide and distilled. Traces of moisture were finally removed azeotropically after adding a small amount (2 ml) of benzene. Isopropyl alcohol (BDH, b.p. 82.5°C) was dried by refluxing over aluminium isopropoxide and distilled. The traces of moisture from the dried isopropyl alcohol were removed by azeotropic fractionation of the resulting ternary azeotrope obtained after addition of 2 ml dry benzene.

Anhydrous $InCl_3$ (Fluka) was sublimed prior to use. Schiff bases were prepared by the condensation reactions of salicylaldehyde and appropriate amine

TABLE 3 NMR Data (δ, ppm) of **1–4**

	$^1H^a$	^{11}B ^{27}Al
1	0.69–0.91 (d, $C\mathbf{H}_3$–CH); 1.04–1.26 (d, $C\mathbf{H}_2$–CH); 3.89–4.18 (sextet, $C\mathbf{H}(CH_3)CH_2)_B$; 6.43–7.54 (m, $C_6\mathbf{H}_4$); 8.02 (s, **H**C=N)	–
2	0.85–1.10 (d, $\mathbf{H}_3C$–CH); 1.20–1.48 (t, $C\mathbf{H}_3$–CH_2); 1.64–1.83 (t, $C\mathbf{H}_3$–CH_2); 3.45–3.80 [sextet, $((CH_3)C\mathbf{H}$–$CH_2)_T$ + quintet, (CH–$C\mathbf{H}_2$–$CH_3)_T$]; 6.37–7.67 (m, C_6H_4); 8.36 (s, C**H**=N)	–
3a	0.50–1.52 [d, $(C\mathbf{H}_3$–CH)/($C\mathbf{H}_2$–CH)/$(C\mathbf{H}_3)_2CH)$]; 2.89–3.48 (septet, $C\mathbf{H}(CH_3)_2)_T$; 3.66–4.12 [septet, $(C\mathbf{H}(CH_3)_2)_B$ + sextet, $(C\mathbf{H}(CH_3)CH_2)_B$]; 6.34–7.48 (m, C_6H_4); 8.30 (s, **H**C=N)	1.8
3b	1.01–1.33 (d, $CH(C\mathbf{H}_3)_2/CHC\mathbf{H}_3/C\mathbf{H}_2CH$); 3.26–3.66 (septet, $C\mathbf{H}(CH_3)_2)_T$; 3.74–4.31 [septet, $(C\mathbf{H}(CH_3)_2)_B$ + sextet,$\mathbf{(CH}(CH_2)CH_3)_B$]; 6.43–7.44 (m, $C_6\mathbf{H}_4$); 8.30 (s, C**H**=N)	38.15
3c	1.01–1.42 (d, $CH(C\mathbf{H}_3)_2/C\mathbf{H}_3$–$CH/C\mathbf{H}_2CH$); 3.29–3.67 (septet, $C\mathbf{H}(CH_3)_2)_T$; 3.77–4.18 [septet, $(C\mathbf{H}(CH_3)_2)_B$ + sextet, $(C\mathbf{H}(CH_3)CH_2)_B$]; 6.43–7.35 (m, $C_6\mathbf{H}_4$); 8.11 (s, C**H**=N)	–
3d	1.07–1.36 (d, $CH(C\mathbf{H}_3)_2/C\mathbf{H}_3CH/CH_2CH$); 3.74–4.18 (septet, $C\mathbf{H}(CH_3)_2)_T$; 4.28–4.96 [septet, $(C\mathbf{H}(CH_3)_2)_B$ + sextet, $(C\mathbf{H}(CH_3)CH_2)_B$]; 6.37–7.70 (m, $C_6\mathbf{H}_4$); 8.17 (s, **H**C=N)	–
4a	1.07–1.45 [d, $CH(C\mathbf{H}_3)_2/(NCH(C\mathbf{H}_3))$ + t, (CH–CH_2–$C\mathbf{H}_3$)]; 3.42–3.80 [quintet, $(C\mathbf{H}_2(CH)CH_3)_T$ + septet, $(C\mathbf{H}(CH_3)_2)_T$]; 3.8–4.31 [septet, $(C\mathbf{H}(CH_3)_2)_B$ + sextet, $(C\mathbf{H}CH_2CH_3)_T$]; 6.40–7.67 (m, $C_6\mathbf{H}_4$), 8.17–8.43 (s, C**H**=N)	3.39
4b	0.91–1.33 [d, $CH(C\mathbf{H}_3)_2/NCH(C\mathbf{H}_3)_2$ + t, $(CH(CH_2)C\mathbf{H}_3)$]; 3.32–3.70 [quintet, $(C\mathbf{H}_2(CH)CH_3)_T$ + septet, $(C\mathbf{H}(CH_3)_2)_T$]; 3.77–4.18 [septet, $(C\mathbf{H}(CH_3)_2)_B$ + sextet, $(C\mathbf{H}CH_2CH_3)_T$]; 6.40–7.44 (m, $C_6\mathbf{H}_4$) 8.02–8.03 (s, C**H**=N)	43
4c	0.92–1.60 [d, $C\mathbf{H}_3CH/NCH(C\mathbf{H}_3)_2$ + t, $(CHCH_2C\mathbf{H}_3)$]; 3.28–3.69 [quintet, $(C\mathbf{H}_2(CH)CH_3)_T$ + septet, $(CH(C\mathbf{H}_3)_2)_T$]; 3.62–4.38 [septet, $(C\mathbf{H}(CH_3)_2)_B$ + sextet, $(C\mathbf{H}(CH_2)CH_3)_T$]; 6.42–7.42 (m, C_6H_4); 8.08–8.26 (s, C**H**=N)	–
4d	0.72–1.52 [d, $C\mathbf{H}_3CH/CH(C\mathbf{H}_3)_2$ + t, $(CHCH_2C\mathbf{H}_3)$]; 3.23–3.67 [quintet, $(C\mathbf{H}_2(CH)CH_3)_T$ + septet, $(C\mathbf{H}(CH_3)_2)_T$]; 3.86–4.88 [septet, $(C\mathbf{H}(CH_3)_2)_B$ + sextet, $(C\mathbf{H}(CH_2)CH_3)_T$]; 6.40–7.35 (m, $C_6\mathbf{H}_4$); 8.14–8.27 (s, C**H**=N)	–

[a] s = singlet, d = doublet, t = triplet, m = multiplet, $_B$ = bridging, $_T$ = terminal, coupling constants for doublet, triplet, quintet, sextet, and septet systems are 5.70 Hz.

TABLE 4 ^{13}C NMR Data (δ, ppm) of **1, 3a–d**

	C–O	*C=N*	*Aromatic Carbon*	*Alkylene Carbon*
1	170.85 169.23	167.59 167.06	133.90–112.29	62.68–63.30 ($\mathbf{C}H(CH_3)CH_2$), 54.47 ($\mathbf{C}H_2$–N), 19.29–18.74 (CH–$\mathbf{C}H_3$)
3a	170.32	166.23	134.54–114.85	65.23 ($\mathbf{C}H(CH_3)_2)_B$; 65.29 ($\mathbf{C}H(CH_3)_2)_T$; 64.20 ($\mathbf{C}H(CH_3)CH_2$); 52.9 ($\mathbf{C}H_2$–N); 29.61 ($CH(\mathbf{C}H_3)_2)_B$; 25.24 ($CH(\mathbf{C}H_3)_2)_T$; 22.57 ($CH\mathbf{C}H_3$)
3b	169.25	163.67	132.78–113.16	65.28 ($\mathbf{C}H(CH_3)_2)_B$; 64.19 ($\mathbf{C}H(CH_3)_2)_T$; 62.95 ($\mathbf{C}H(CH_3)CH_2$); 56.61 ($\mathbf{C}H_2$–N); 29.90–28.69 ($CH(\mathbf{C}H_3)_2)_B$; 24.26 ($CH(CH_3)_2)_T$; 21.85 (CH–$\mathbf{C}H_3$)
3c	170.67–169.70	162.88	134.07–113.32	65.29 ($\mathbf{C}H(CH_3)_2)_B$; 63.52 ($\mathbf{C}H(CH_3)_2)_{ax}$; 63.02 ($\mathbf{C}H(CH_3)_2)_{eq}$; 62.78 ($\mathbf{C}H(CH_3)CH_2$); 54.95–53.42 ($\mathbf{C}H_2$–N); 28.59 ($CH(\mathbf{C}H_3)_2)_B$; 24.62–24.23 ($CH(\mathbf{C}H_3)_2)_{ax+eq}$; 20.34 (CH–$\mathbf{C}H_3$)
3d	168.00	158.25	131.70–112.00	69.90 ($\mathbf{C}H(CH_3)_2)_B$; 63.97 ($\mathbf{C}H(CH_3)_2)_T$; 58.65 ($\mathbf{C}H(CH_3)CH_2$); 54.68 ($\mathbf{C}H_2$–N); 29.72–28.85 ($CH(\mathbf{C}H_3)_2)_B$; 22.78–22.16 ($CH(\mathbf{C}H_3)_2)_T$; 20.96–20.62 ($CH(\mathbf{C}H_3)$)

$_B$ = bridging, $_T$ = terminal, $_{ax}$ = axial, $_{eq}$ = equitorial.

[7]. Isopropoxides of boron [8], aluminium [9], titanium [11], and niobium [10] as well as potassium isopropoxymetallates of boron, aluminium [12,13], titanium [14], and niobium [15] were prepared according to the literature procedures. Boron, aluminium, and indium were determined by the methyl borate and oxinate methods [16] respectively. Titanium and niobium were determined by metal oxide method [16]. Nitrogen and chloride were determined by Kjeldahl's and Volhard's method, respectively. ^{1}H (89.55 MHz) ^{11}B (28.69 MHz), ^{27}Al (23.2 MHz), and ^{13}C (300 MHz) NMR spectra in $CDCl_3$ solution were recorded on JEOL FX 90Q and on Bruker DPX 300 MHz spectrometers. IR spectra were recorded as Nujol mulls on a Nicolet Magna 550 spectrophotometer using CsI optics. Molecular weights were determined ebullioscopically in benzene using a Gallenkamp ebulliometer.

Preparation of 1

Potassium 0.41 g (10.7 mg atom) was added to the benzene solution of ethyl alcohol and heated till it reacted completely. Then 0.96 g (5.35 mmol) of L^1H_2 was added. After refluxing the reaction mixture for 4 h, a benzene solution of $InCl_3$ 1.18 g (5.35 mmol) was added. The resulting reaction mixture was initially stirred at room temperature for 4 h, followed by refluxing for 6 h. The precipitated KCl 0.79 g (10.59 mmol) was filtered out. Removal of the volatiles from the filtrate under reduced pressure afforded **1** 1.50 g (88%), which was recrystallized from a 1:2 mixture of benzene and *n*-hexane at 0°C to obtain analytically pure product in 1.25 g (75%) yield.

Adopting a procedure similar to that for **1**, the reaction of 1.10 g (4.96 mmol) $InCl_3$ with 1.48 g (9.92 mmol) L^2H in the presence of two equivalents of KOEt, prepared by the interaction of potassium (0.39 g, 9.92 mg atom) with ethyl alcohol, afforded **2**.

Preparation of 3a

Freshly prepared potassium tetraisopropoxyborate (0.44 g, 1.56 mmol) was mixed with a benzene

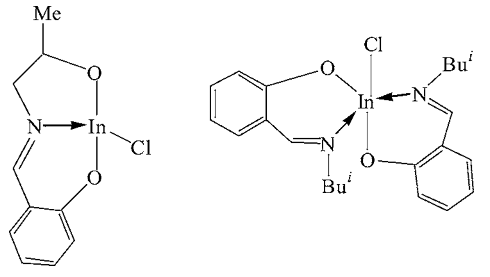

FIGURE 1 Proposed structure of **1** and **2**.

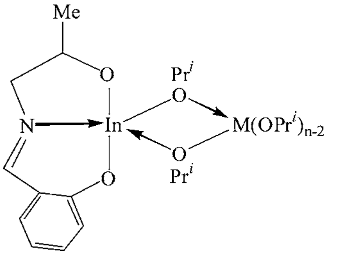

FIGURE 2 Proposed structure for **3**.

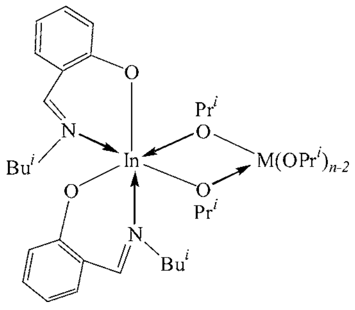

FIGURE 3 Proposed structure of **4**.

solution of $In(L^1)Cl$ (0.51 g, 1.56 mmol), and the resulting reaction mixture after stirring at room temperature for 4 h was finally refluxed for 6 h. The precipitated KCl (0.12 g, 1.60 mmol) was removed by filtration. The removal of volatile components from filtrate under reduced pressure afforded 0.78 g (93%) **3a**, which was recrystallized from a 1:2 mixture of benzene and *n*-hexane at 0°C to obtain the analytically pure compound in 86.72% (0.72 g) yield.

Adopting a procedure similar to that for **3a** derivatives, **3b–d** and **4a–d** were prepared from appropriate reactants. Amounts of the reactants actually used are shown in brackets.

3b: $KAl(OPr^i)_4$ (0.84 g, 2.77 mmol) and $In(L^1)Cl$ (0.91 g, 2.27 mmol)

3c: $KTi(OPr^i)_5$ (0.62 g, 1.62 mmol) and $In(L^1)Cl$ (0.53 g, 1.62 mmol)

3d: $KNb(OPr^i)_6$ (0.62 g, 1.28 mmol) and $In(L^1)Cl$ (0.42 g, 1.28 mmol)

4a: $KB(OPr^i)_4$ (0.45 g, 1.59 mmol) and $In(L^2)_2Cl$ (0.71 g, 1.59 mmol)

4b: $KAl(OPr^i)_4$ (0.57 g, 1.89 mmol) and $In(L^2)_2Cl$ (0.85 g, 1.89 mmol)

4c: $KTi(OPr^i)_5$ (0.64 g, 1.67 mmol) and $In(L^2)_2Cl$ (0.74 g, 1.67 mmol)

4d: $KNb(OPr^i)_6$ (0.61 g, 1.25 mmol) and $In(L^2)_2Cl$ (0.56 g, 1.25 mmol)

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