Chiral base-mediated benzylic functionalisation of (alkyl benzyl ether)tricarbonylchromium(0) complexes: a structure–reactivity study

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The high-yielding readily-analysed conversion of (benzyl methyl ether)tricarbonylchromium(0) **1** into its derivative tricarbonyl[α-(phenylsulfanyl)benzyl methyl ether]chromium(0) **3** has been used to examine the effectiveness of a range of non-racemic chiral lithium amide bases. The most selective base, **2d**, was studied in greater detail to determine the effect of changes to structure, temperature and stoichiometry on its activity.

Non-racemic chiral lithium amide bases are now well-established as useful reagents for asymmetric synthesis.[1] Although to date the area is dominated by work on enantioselective deprotonation of ketones and the enantioselective rearrangement of epoxides to allylic alcohols, a significant number of studies have been carried out on other reactions including enantioselective *aromatic* functionalisation of (arene)-tricarbonylchromium(0) complexes.[2] Recently we reported that *benzylic* functionalisation of (arene)tricarbonylchromium(0) complexes can be achieved using a chiral lithium amide base;[3] specifically, the benzylic methylene group in complexes of alkyl benzyl ethers such as **1** can be functionalised asymmetrically in high yield and enantiomeric excess using the non-racemic chiral lithium amide base **2d**.[3] Subsequent development of this work has led to the synthesis of tertiary benzyl ether complexes of high enantiomeric purity,[4] asymmetric functionalisation of complexes of benzyl sulfides,[5] a highly enantioselective chiral base mediated [2,3]-Wittig rearrangement,[6] and a new approach to non-racemic *N*-hydroxycarbamates and amines.[7] In parallel, Simpkins and co-workers have demonstrated the asymmetric benzylic functionalisation of the tricarbonylchromium(0) complexes of 1,3-dihydroisobenzofuran[8] and 1,3-dihydroisobenzothiophene.[9]

The structure of the lithium amide base and the reaction conditions used in an asymmetric functionalisation have a profound effect on the enantioselectivity of the reaction.[1] We present herein a detailed account of our original study with base **2d** and tricarbonylchromium(0) complexes of alkyl benzyl ethers,[3] together with a new investigation into how base structure and reaction conditions affect the enantioselectivity of the functionalisation.

Results and discussion

Deprotonation of (benzyl methyl ether)tricarbonylchromium(0) **1** and subsequent quenching with electrophiles is well established as an efficient process.[10] In view of the success achieved in the differentiation between enantiotopic aromatic hydrogens in (arene)tricarbonylchromium(0) complexes,[2] it was of interest to us to determine whether or not the enantiotopic benzylic hydrogens in **1** could be replaced stereoselectively using a non-racemic chiral lithium amide base followed by an electrophilic quench. If this were the case, it would demonstrate for the first time that benzylic hydrogens of (arene)tricarbonylchromium(0) complexes may be differentiated by chiral bases. Our initial choice of base was the well-established lithium amide base **2a** which gives high levels of selectivity in *inter alia* enantioselective ketone deprotonations and aromatic functionalisations of (arene)tricarbonylchromium(0) complexes.[1]

Table 1 Reactions of non-racemic chiral lithium amides **2** with ether complex **1** to give phenylsulfanyl derivative **3**

Entry	Amide	Yield (%)	Ee (%)	*R*/*S*	$[\alpha]_D$[a]
1	**2a**[b]	52	22	*S*	+17
2	**2b**[b]	87	0	—	—
3	**2c**[b]	99	29	*S*	+22
4	**2d**[c]	95	98	*R*	−79
5	**2e**[d]	58	10	*R*	−7
6	**2f**[d]	83	49	*S*	+22
7	**2g**[d]	55	27	*S*	+19
8	**2h**[d]	*e*	—	—	—
9	**2i**[c]	93	32	*R*	−23
10	**2j**[c]	88	5	*S*	+5
11	**2k**[d]	80	40	*R*	−23

[a] All measurements were made in CH_2Cl_2 at $c = 1$ in the temperature range 25–31 °C. [b] Reaction carried out using 2.2 equiv. of Bu^{n}Li, 1.1 equiv. of amino·HCl without addition of LiCl (see general procedure in Experimental section). [c] Reaction carried out using 2.2 equiv. of Bu^{n}Li, 1.1 equiv. of diamine and 1 equiv. of LiCl (see general procedure). [d] Reaction carried out using 1.1 equiv. of Bu^{n}Li, 1.1 equiv. of amine and 1 equiv. of Li Cl (see general procedure). [e] 98% recovery of **1**.

(Benzyl methyl ether)tricarbonylchromium(0) **1**[10] and the amine[11] corresponding to amide **2a** were synthesised from benzyl alcohol and (*R*)-α-methylbenzylamine respectively using literature methods. In our first experiment, chiral base **2a** was used to deprotonate **1** and diphenyl disulfide was used to quench the reaction. Work-up gave the novel α-(phenylsulfanyl)benzyl methyl ether complex **3** in 52% yield. The product of this reaction was readily analysed by chiral HPLC and its ee was found to be a moderate 22% (Table 1, entry 1). Nevertheless, we were encouraged by the fact that some discrimination had occurred, and chose to continue our investigation using the established chiral bases **2b**, **2c** and the relatively underemployed diamide **2d**, the precursor amines of which were readily synthesised using literature methods starting from (*R*)-α-methylbenzylamine and 2-bromopropane,[12] (+)-camphor[13] and glyoxal[14] respectively. The reaction of amide **2b**, containing just

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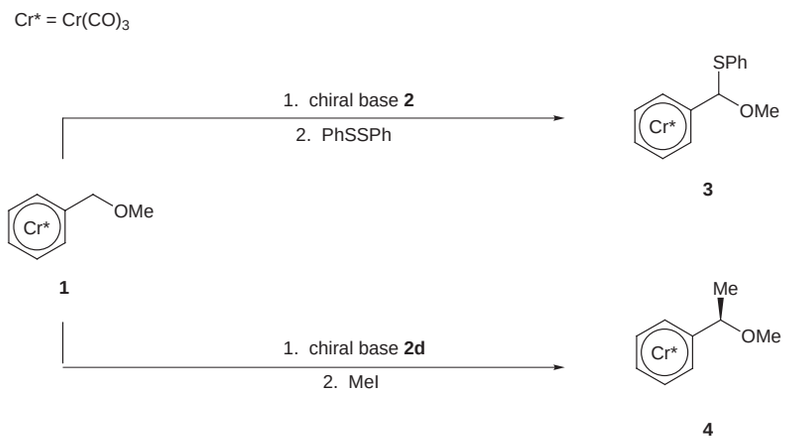

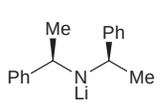

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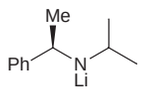

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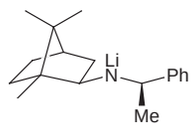

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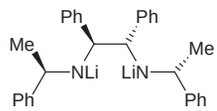

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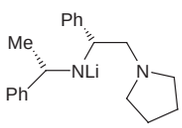

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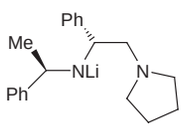

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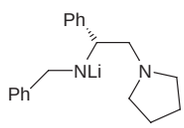

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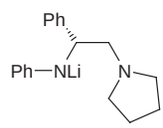

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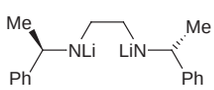

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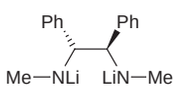

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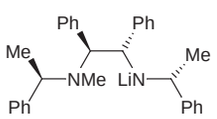

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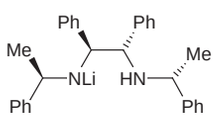

2d'

one chiral centre, with complex **1** followed by the diphenyl disulfide quench gave product **3** in a disappointing 0% ee (Table 1, entry 2), whilst the camphor-based amide **2c** gave an ee of 29% (Table 1, entry 3). In contrast, and to our great delight, the diamide **2d** led to **3** in 98% ee and a respectable 95% yield (Table 1, entry 4).

In order to include a second category of non-racemic chiral lithium amide base[1] in our screen for enantioselectivity, and in view of the general level of success achieved with vicinal diamines in a wide range of enantioselective processes,[15] we elected to study bases **2e**–**h**, the parent amines of which are readily available from (*R*)-styrene oxide, pyrrolidine and the appropriate primary amine *via* a one-pot synthesis.[16] Under our standard conditions, **2e**, **2f** and **2g** gave **3** in 10, 49 and 27% ee respectively (Table 1, entries 5–7) whilst the aniline derived base **2h** failed to produce **3** (Table 1, entry 8).

Thus, from our study of bases **2a**–**2h**, base **2d** was clearly the most effective for the enantioselective conversion of **1** to **3** and it was decided to study this base in more detail. Beforehand, however, we needed to determine the absolute configuration of the products of our reactions. This was achieved by examining a related reaction in which complex **1** was reacted with chiral base **2d** and then quenched with iodomethane. This gave the 1-phenylethyl methyl ether complex **4** in 96% yield and 97% ee. Comparison of the $[a]_D$ of this material with literature data[17] revealed that the absolute configuration of **4** was *R*. Thus, assuming that the diphenyl disulfide quench and the iodomethane quench are stereochemically identical, then the product of the reaction involving complex **1** and base **2d** (Table 1, entry 4) must be the complex **3** of *R* configuration. The stereochemistry of the samples of complex **3** produced in all the other reactions involved in this study was determined by comparision of their optical rotation and HPLC data with this reaction.

In view of the remarkable enantioselectivity observed using diamide **2d**, it was decided to examine bases which carried just some of the structural features of **2d** in order to determine whether or not all of the components of the base were necessary for high enantioselectivity. Thus the parent amines of bases **2i**, **2j** and **2k** were synthesised by reaction of (*R*)-α-methylbenzylamine with dichloroethane,[18] reductive imine coupling followed by resolution with L-(+)-tartaric acid,[19] and *N*-methyl-

Table 2 Effect of temperature on the reaction between non-racemic homochiral lithium amide **2d** and ether complex **1** to give phenylsulfanyl derivative **3**[a]

Entry	*T*/°C	Yield (%)	Ee (%)	*R*/*S*	$[\alpha]_D$[b]
1	−78	95	98	*R*	−79
2	−23	87	93	*R*	−65
3	20	*c*			

[a] All reactions were carried out using 2.2 equiv. of BuLi and 1.1 equiv. of diamine. The temperature given is that of the reaction between **2d** and **1**. The quench was carried out in the standard manner (see general procedure in Experimental section). [b] All measurements were made in CH_2Cl_2 at $c = 1$ in the temperature range 25–31 °C. [c] Mixture of products with no evidence for formation of complex **3**.

Table 3 Effect of varying the stoichiometry of BuLi and the diamine precursor to **2d** on the reaction between non-racemic chiral lithium amide **2d** and ether complex **1**[a]

Entry	BuLi (equiv.)	Diamine (equiv.)	Yield **3** (%)	Ee (%)	Recovered **1** (%)
1	2.2	1.1	95	98	0
2	1.1	1.1	96	95	0
3	0.55	1.1	60	92	40
4	0.275	1.1	21	96	67
5	1.1	0.55	88	96	0
6	1.1	0.275	64	73	10

[a] Stoichiometries are given relative to ether complex **1**. All reactions were performed in the presence of 1 equiv. of LiCl and quenched with 2 equiv. of diphenyl disulfide. The absolute configuration of the product was *R* in all cases.

ation of **2d**[20] respectively. The use of bases **2i**–**k** for the conversion of complex **2** to complex **3** gave enantioselectivities of 32, 5 and 40% respectively. Thus removal of the backbone phenyl groups from **2d**, removal of its side-arm chirality, and replacement of one of its lithium amide sites with a tertiary amine all significantly reduce its effectiveness.

Having established that the readily synthesised non-racemic chiral lithium amide base **2d** was the most effective reagent for asymmetric functionalisation of complex **1**, we elected to probe the effects of both temperature and stoichiometry on the reaction. Using otherwise identical conditions, the reaction between **2d** and **1** followed by a diphenyl disulfide quench was carried out at −78, −23 and 20 °C. The results from the reactions performed at −78 and −23 °C were essentially identical (Table 2, entries 1 and 2), indicating that the benzylic anion generated in the reaction retains stereochemical information even at relatively high temperatures, whereas at 20 °C, a complicated mixture of products was formed, presumably due to the chemical instability of the anion at this temperature.

All the reactions discussed above were carried out using 2.2 equiv. of BuLi and 1.1 equiv. of diamine relative to complex **1**. In an effort to minimise the amount of BuLi and diamine required, a set of experiments were carried out which used varying quantities of these two reagents. Initially the amount of diamine used was held constant at 1.1 equiv. and the amount of BuLi added reduced from 2.2 equiv. to 1.1, 0.55 and finally 0.275 equiv. The results from these experiments (Table 3, entries 1–4) indicate that 1.1 equiv. of BuLi is essentially as effective as 2.2 equiv., and that whilst lower amounts of BuLi lead, not surprisingly, to lower yields, the enantioselectivity of the reaction remains high. Next, the equivalence of BuLi was held constant at 1.1, whilst the equivalence of diamine was reduced from 1.1 to 0.55 and finally 0.275 (Table 3, entries 2, 5 and 6). Whilst 1.1 and 0.55 equiv. of diamine gave essentially identical results, the experiment performed with 0.275 equiv. gave a poor yield and ee indicating that the reaction is not catalytic with respect to this diamine.

The results in Table 3 may be explained as follows. The dilithiated species **2d** (or an aggregate derived from **2d**) is clearly a highly enantioselective reagent as illustrated by entry 1. If we assume that the monolithiated species **2d′** is relatively unselective, a hypothesis supported to a degree by the low ee (40%) obtained with **2k**, then it follows from the results in Table 3 that i) **2d** is more reactive than **2d′** with respect to complex **1**, and ii) there is a ready exchange of lithium cations between all the nitrogen sites in the system. If these two conditions were not met then the ees obtained in entries 2–5 would be significantly diminished with respect to entry 1. The reduced ee obtained in entry 6 is explained by a high yielding highly enantioselective reaction driven by half of the BuLi used (funnelled through **2d**), and a low yielding (approximately 50%—see Experimental section for synthesis of racemic **3**) non-enantioselective direct deprotonation of **1** by the remaining BuLi.

In conclusion we have used the high-yielding, easily-analysed conversion of complex **1** into its derivative **3** to examine the selectivity of a range of non-racemic chiral lithium amide bases. The most selective system **2d** was examined in more detail with respect to structure, temperature and stoichiometry, studies which have provided some insight into its mode of action.

Experimental

All reactions and manipulations were performed with the exclusion of light under nitrogen using standard vacuum line and Schlenk tube techniques.[21] Tetrahydrofuran was distilled from sodium benzophenone ketyl. The concentrations of alkyllithiums were determined by titration against diphenylacetic acid in THF.[22] Complex **1**[10] and the amine precursors to **2a**,[11] **2b**,[12] **2c**,[13] **2d**,[14] **2e**–**h**,[16] **2i**,[18] **2j**[19] and **2k**[20] were prepared according to literature methods. All other reagents were used as obtained from commercial sources, unless otherwise stated.

Melting points were determined in sealed capillaries under nitrogen using either an Electrothermal IA9100 digital or Büchi 510 melting point apparatus, and are uncorrected. IR spectra were obtained on Perkin-Elmer 1710 or Mattson 5000 FTIR spectrometers. Elemental analyses were performed by Imperial College Microanalytical Service. Optical rotations were recorded on a Perkin-Elmer 241 polarimeter with a 10 cm path length. Concentrations for optical rotation measurements are given in g 100 cm^{-3}. NMR spectra were recorded at room temperature on JEOL GSX 270 (270 MHz ^1H, 67.9 MHz ^{13}C) or Bruker DRX 300 (300 MHz ^1H, 75.4 MHz ^{13}C) instruments. ^1H NMR chemical shifts are referenced with respect to residual undeuterated solvent while ^{13}C NMR shifts are referenced with respect to deuterated solvent. Broadband ^1H decoupling was employed for ^{13}C NMR spectra. *J* values are given in Hz. Mass spectra were recorded on VG Micromass 7070E or Autospec-Q instruments at Imperial College using EI techniques. Analytical HPLC was carried out using a Unicam Crystal 200 pump with a Unicam Spectrea 100 UV-vis detector at Imperial College.

Synthesis of racemic tricarbonyl[η^6-α-(phenylsulfanyl)benzyl methyl ether]chromium(0) 3

A stirred solution of complex **1** (100 mg, 0.39 mmol) in THF (2.5 cm^3) at −78 °C was treated with Bu^{t}Li (1.3 M in pentane, 0.36 cm^3, 0.47 mmol). After 20 min, a solution of PhSSPh (120 mg, 0.55 mmol) in THF (1 cm^3) was added and the mixture stirred at −78 °C for 0.5 h before addition of methanol (1 cm^3) and warming to room temperature. The solvent was removed *in vacuo* and the residue subjected to flash chromatography [Al_2O_3, basic, Brockmann II; diethyl ether–petroleum ether (bp 40–60 °C), 1:7], followed by recrystallisation from pentane (crystallisation at −78 °C) to give the *title complex* **3** as a yellow crystalline solid (68 mg, 0.186 mmol, 48%), mp 91.5–92 °C (Found: C, 55.45; H, 3.9. $C_{17}H_{14}CrO_4S$ requires C, 55.74; H, 3.85%); ν_{max} (CH_2Cl_2)/cm^{-1} 1968s and 1887s (C≡O); δ_H (300

MHz; $CDCl_3$) 3.69 (3 H, s, CH_3), 4.99 (1 H, m, Cr-C*H*), 5.13–5.17 (2 H, m, Cr-C*H*), 5.26 (1 H, s, C*H*OMe), 5.32 (2 H, m, Cr-C*H*) and 7.25–7.33 (5 H, m, Ph); δ_C (67.9 MHz; $CDCl_3$) 56.9 (*C*H_3), 88.8, 89.5, 90.1, 91.0, 91.9 and 92.4 (Cr-$C_{ortho/meta/para}$ and *C*HOMe), 110.6 (Cr-C_{ipso}), 128.7 and 128.8 (Ph-$C_{ortho/meta/para}$), 135.9 (Ph-C_{ipso}) and 232.8 (*C*≡O); *m*/*z* (EI) 366 (M^+, 6%), 282 (M − 3CO, 22), 167 [$(Ph)_2CH$, 100] and 52 (Cr, 63).

Synthesis of racemic tricarbonyl(η⁶-1-phenylethyl methyl ether)chromium(0) 4[10]

To a stirred solution of complex **1** (100 mg, 0.39 mmol) in THF (5 cm^3) at −78 °C was added Bu^{t}Li (1.7 M in pentane, 0.25 cm^3, 0.43 mmol). The solution was stirred at −78 °C for 15 min after which MeI (56 μl, 0.90 mmol) was added. The temperature was maintained at −78 °C for 0.5 h, methanol (1 cm^3) was added and the mixture was allowed to warm to room temperature. The solvent was removed *in vacuo* and the residue subjected to flash chromatography [SiO_2; diethyl ether–petroleum ether (bp 40–60 °C), 1 : 4], followed by recrystallisation from pentane to give the title complex **4** as a yellow crystalline solid (95 mg, 0.35 mmol, 90%), mp 54–55 °C (lit.[17] mp 57 °C); ν_{max} (CH_2Cl_2)/cm^{-1} 1968s and 1889s (C≡O); δ_H (300 MHz; $CDCl_3$) 1.46 (3 H, d, *J* 6.5, CHCH_3), 3.47 (3 H, s, OCH_3), 4.06 (1 H, q, *J* 6.5, OC*H*), 5.33–5.37 (4 H, m, Cr-C*H*) and 5.53 (1 H, m, Cr-C*H*); δ_C (75.4 MHz; $CDCl_3$) 22.2 (CH*C*H_3), 57.3 (O*C*H_3), 76.9 (O*C*H), 90.8, 91.7 and 92.6 (Cr-$C_{ortho/meta/para}$), 113.4 (Cr-C_{ipso}) and 232.9 (*C*≡O); *m*/*z* (EI) 272 (M^+, 3%), 188 (M − 3CO, 5), 156 (M − 3CO–CH_3OH, 12), 91 ($PhCHCH_3$, 24), 77 (Ph, 35) and 52 (Cr, 100).

General procedure for reaction of non-racemic chiral lithium amide 2 with ether complex 1 to give phenylsulfanyl derivative 3. (Tables 1–3)

A solution of lithium (di)amide was prepared by addition of the required amount of Bu^{n}Li (1.6 M in hexanes) (see Tables for equivalence relative to **1**) to a stirred solution of chiral (di)amine or amine·HCl (see Tables for equivalence relative to **1**) in THF (5 cm^3) at −78 °C, and the solution was allowed to warm to room temperature. The resulting pink solution was recooled to −78 °C and a solution of flame-dried LiCl (17 mg, 0.40 mmol, 1 equiv.) in THF (5 cm^3) was added *via* a cannula. After 5 min a precooled (−78 °C) solution of complex **1** (0.103 g, 0.40 mmol, 1 equiv.) in THF (5 cm^3) was added dropwise *via* a short cannula over approximately 2 min. The orange–yellow solution was stirred at −78 °C for 0.5 h, after which the electrophile diphenyl disulfide (175 mg, 0.80 mmol, 2 equiv.) was added. After a further 0.5 h at −78 °C, MeOH (1 cm^3) was added, the mixture allowed to warm to room temperature and the solvents removed *in vacuo*. The yellow residue was purified by flash chromatography [Al_2O_3, basic, Brockmann II; diethyl ether–petroleum ether (40–60 °C), 1 : 4] and the product identified by ^{1}H NMR spectroscopy and TLC, which were identical to that of an authentic sample of the racemic complex. HPLC: Chiralcel OD-H column; eluent propan-2-ol–hexane, 1 : 19; flow rate 1 cm^3 min^{-1}; detection 330 nm; (+)-**3** RT 14.5 ± 2 min, (−)-**3** RT 18.5 ± 3 min.

Synthesis of (+)-tricarbonyl(η⁶-1-phenylethyl methyl ether)-chromium(0) 4 using non-racemic chiral lithium amide 2d

Following the general procedure described above, BuLi (1.6 M, 0.54 cm^3, 0.86 mmol) was added to the diamine precursor to **2d** (180 mg, 0.43 mmol) in THF (5 cm^3). LiCl (17 mg, 0.40 mmol) in THF (5 cm^3) was added, followed by complex **1** (100 mg, 0.39 mmol) in THF (5 cm^3) and after 1 h, MeI (0.10 cm^3, 1.6 mmol). MeOH was added after 1 h and, following flash chromatography [SiO_2; diethyl ether–petroleum ether (bp 40–60 °C), 1 : 4] the title complex **4** was obtained as a yellow crystalline solid (101 mg, 0.37 mmol, 96%). The product was identified by ^{1}H NMR spectroscopy and TLC, which were identical to that of an authentic sample of the racemic complex. $[\alpha]_D^{26}$ +54 (*c* = 1, CH_2Cl_2); HPLC: Chiralcel OD-H column; eluent propan-2-ol–hexane, 1 : 39; flow rate 1 cm^3 min^{-1}; detection 330 nm; (−)-**4** RT 16.7 min, (+)-**4** RT 18.8 min; ee 97%. A sample (79 mg, 0.29 mmol) was recrystallised from pentane to give the title complex as yellow needles (67 mg, 85% crystallisation yield); $[\alpha]_D^{27}$ +53 (*c* = 1, CH_2Cl_2); ee 98.5%.

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