with degassed 3M KOH (10 mL). The mixture was stirred at RT for 30 min. The two layers were then separated and the organic phase dried ($MgSO_4$) under argon. The resulting clear yellow organic phase was filtered and transferred by cannulation into a second flask flushed with argon. The toluene was evaporated and the crude diphosphane **7d** washed with dried degassed methanol (2×5 mL). After filtration, traces of the solvent were removed under high vacuum (2 h) to yield **7d** as a white microcrystalline solid (m.p. 126–128 °C), which was stored under argon (0.76 g, 80 % yield).

Typical procedure for the hydroboration with **7d**: A mixture of $[Rh(cod)_2]BF_4$ (8.1 mg, 0.020 mmol) and diphosphane **7d** (11.5 mg, 0.024 mmol) in dry DME (5 mL) was stirred for 10 min at RT in a 10-mL Schlenk tube under argon. Styrene or a derivative (2 mmol) was added to the resulting orange solution. The homogeneous mixture was cooled to $-35\,^{\circ}C$ and stirred at this temperature for 15 min before adding freshly distilled catecholborane dropwise (2.4 mmol, 0.26 mL). The catecholborane dissolved in the DME and some gas evolved from the reaction mixture. The reaction was monitored by sampling. Aliquots were taken, treated with KOH (3M) and 30 % H_2O_2, and extracted with diethyl ether or dichloromethane. The samples were then analyzed by chiral GC (Chiralsil DEX-CB column) or chiral HPLC (OD or OJ columns) to determine the conversion (using *n*-decane as an internal reference) and enantiomeric excess. The regioselectivity was determined by ^1H NMR spectroscopic analysis of the final crude reaction mixture after oxidative work-up. The products were purified by chromatography on silica (pentane/diethyl ether) to afford the corresponding alcohols.

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The First Crystallographic Evidence for the Structures of *ortho*-Lithiated Aromatic Tertiary Amides**

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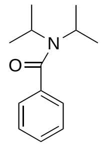
Directed metalation is arguably the most selective way of making regiospecifically substituted aromatic rings. The use of directed *ortho*-metalation involving amide-type functional groups (secondary and tertiary amides, carbamates, and oxazolines) has revolutionized the synthesis of complex benzenoid aromatic compounds over the last 15 years:[1] several recent total syntheses have involved important *ortho*-lithiation steps.[2] The four classes of substituents mentioned are not only the best directors of lithiation—with their electron-rich oxygen centers which promote the "complex-induced proximity effect"[3] and withdraw electron density from the ring—but also the most versatile.[2c]

It is, therefore, surprising how little is known of the products of *ortho*-lithiation reactions. Kinetic-isotope-effect evidence[4] suggests the reaction proceeds by a rate-determining deprotonation of an initial substrate–organolithium complex. It is assumed that O–Li coordination is maintained from reactive complex through to products, though for tertiary amides (the best directing group of all[5]) such coordination poses severe geometric difficulties. Even in the simple benzamide **1**, the tertiary amide group lies twisted out of the aromatic ring plane for steric reasons,[6, 7] inhibiting direct O-coordination to a 2-lithio group. The angle of twist affects the rate of lithiation,[6] but even amides which have little flexibility to rotate far from perpendicular, for example, **2** and **3**, still undergo efficient *ortho*-lithiation.[7, 8]



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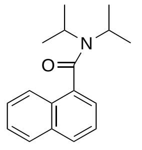

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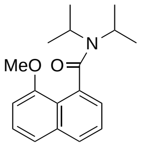

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Herein we report the first crystal structures of the products of tertiary-amide-directed *ortho*-metalation reactions. These go some way towards clarifying the nature of the O–Li coordination in *ortho*-lithiated amides, and also towards

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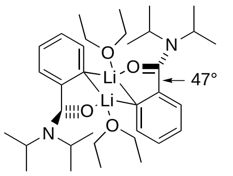
explaining the surprising ease with which hindered tertiary amides may be *ortho*-lithiated, despite their twisted geometry.

Treatment of *N*,*N*-diisopropylbenzamide **1** in diethyl ether at −78 °C with one equivalent of *t*BuLi gave a yellow solution. On storage at −30 °C, crystals were formed which were identified by NMR spectroscopy (see Experimental Section) as being of an *N*,*N*-diisopropyl-2-lithiobenzamide-diethyl ether complex **4**. The X-ray structure[9] of these crystals showed that in the solid state **4** is a dimer in which an (LiC)$_2$ core is stabilized by the O-centers of the amide carbonyl functionalities and of Et$_2$O (Figure 1).



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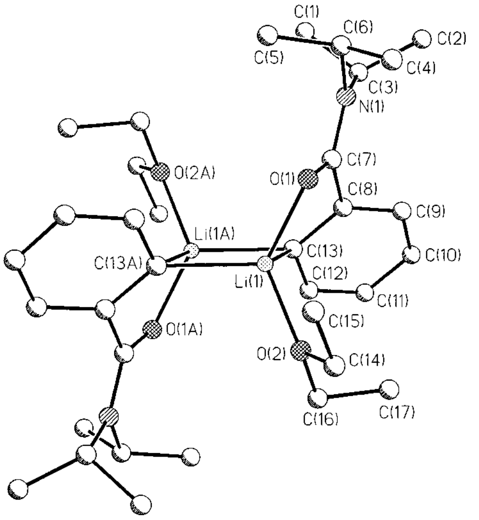
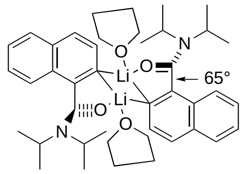


Figure 1. Molecular structure of **4**; hydrogen atoms and disorder in the Et$_2$O molecules are omitted for clarity.

An (LiX)$_2$ unit is not uncommon in species of the type [(RLi·L)$_n$] in which R is an imide (X = N),[10] alkoxide,[11] enolate[12] (X = O), or aryl (X = C)[13] residue and L is a monodentate Lewis base (THF, Et$_2$O etc.). The Li–C bonds in the core of **4** vary in a fashion explicable by viewing the structure as an aggregate of two monomeric organolithium fragments with their metal centers stabilized by intramolecular coordination to the oxygen atom of the amide group (Li(1)–O(1) = 1.936(3) Å). Each lithium ion lies close to the plane of the amide system (dihedral angle N(1)-C(7)-O(1)-Li(1) = 161.1°), which is itself twisted at an angle of 46.8° to the aromatic ring. The strain imposed by this twist means that the intramolecular C–Li bond is long and weak (Li(1)–C(13) = 2.303(4) Å) and that the lithium center lies well out of the plane of the aromatic ring. Dimerization allows the formation of a stronger metal–carbon bond (Li(1)–C(13A) = 2.176(4) Å) lying surprisingly close to the aromatic plane (dihedral angle C(7)-C(8)-C(13)-Li(1A) = 21.0°).

In a similar way, reaction of *N*,*N*-diisopropyl-1-naphthamide **2** in THF at −78 °C with one equivalent of *t*BuLi gave a yellow suspension which dissolved at reflux. Storage of the resultant solution at −30 °C afforded yellow crystals of the *N*,*N*-diisopropyl-2-lithionaphthamide-THF complex **5** (see Experimental Section). The X-ray structure[14] revealed that in the solid state **5** is also a dimer (for which there are two molecules of lattice THF) in which an (LiC)$_2$ core is stabilized by amide groups and O centers from THF (Figure 2). As with **4**, the structure of **5** suggests that dimerization accompanies the intramolecular amide-group coordination of each lithium center (Li(1)–O(1) = 1.974(4) Å). The significant lengthening of this bond as compared with its analogue in **4** is accompanied by lengthening of the intramolecular C–Li bond (Li(1)–C(8) = 2.389(4) Å). Relative to **4**, these changes result from the even greater 65° dihedral angle between the amide group and the aromatic ring in **5**. The amide group and ring plane in naphthamides and in 2-substituted benzamides typically lie almost perpendicular,[6, 7] and the 65° dihedral angle must represent a compromise between maintaining intramolecular C-Li-O bonding and minimising steric hindrance.



5

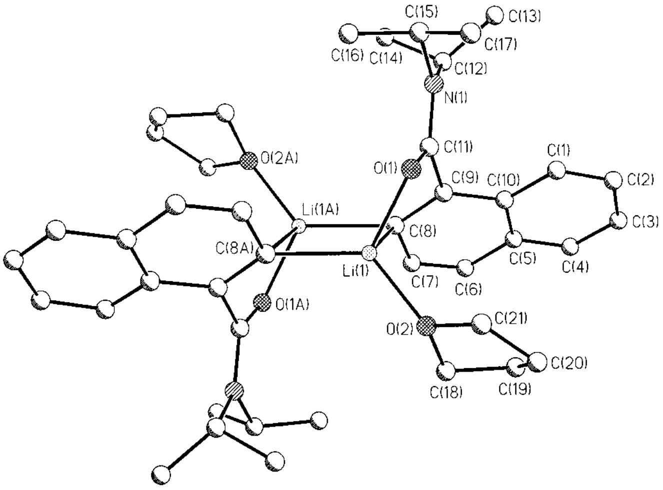


Figure 2. Molecular structure of **5**; hydrogen atoms and disorder in the THF molecules are omitted for clarity.

The solid-state structures of both **4** and **5** establish for the first time that the oxygen coordination of the lithium center is maintained in *ortho*-lithiated aromatic tertiary amides, despite the deviation from coplanarity[6, 7] of the amide group and the aryl system in precursors **1** and **2**. The structures show that the formation of a four-membered (LiC)$_2$ ring is ideally suited to stabilization of the metal by the amide carbonyl group, but the increased strain evident in the lengthened bonds of **5** accounts for the slower rates of lithiation of these twisted amides relative to their coplanar analogues.[6]

Complexes **4** and **5** represent rare examples of β-lithiated amides.[15] Solid-state structures of *ortho*-lithiated benzenoid derivatives have been reported with a variety of *saturated* directing groups[16] but the crystal structures of **4** and **5** are the first to incorporate *ortho*-metalated carbonyl-substituted aromatic rings. *Ortho*-lithiated compounds containing *unsaturated* directing groups have been characterized only for polymetalated stilbenes,[17] sulfones,[18] and sulfoximines.[19]

In conclusion, the isolation and characterization of **4** and **5** afford a significant insight into the *ortho*-lithiation-directing effects of tertiary amides. Complexation of the lithium center by the amide C=O group, as expected, is crucial to the stability of the metalated species, and this mode of coordination is achieved by lessening the amide–arene twist angle. Solution studies on both **4** and **5** will form the basis of future work with preliminary NMR spectroscopy pointing to rather complicated solution behavior. Hence **5** dissolves in [D$_8$]THF to give five observable solution species (#1–#5, see Experimental Section). Further, we intend, by the use of theoretical methods, to probe the mechanism by which directed *ortho*-lithiation occurs and also hope to extend the study to the metalation of other, more complex, atropisomeric tertiary amides with a view to gaining a better understanding of the stereochemistry of these unusual chiral organolithium species and their reactions.[20]

Experimental Section

4: *t*BuLi (0.8 mL, 1.7 M in pentane, 1.3 mmol) was added to a solution of *N*,*N*-diisopropylbenzamide **1** (0.26 g, 1.25 mmol) in Et$_2$O (10 mL) under nitrogen at −78 °C. The resultant yellow solution was transferred directly to a −30 °C freezer. Yellow crystals of **4** were obtained after 4 h at this temperature. Yield 65 %; m.p. 101 °C (decomp.); elemental analysis calcd (%) for $C_{17}H_{28}LiNO_2$: C 71.56, H 9.89, N 4.91; found: C 71.29, H 9.89, N 5.10; ^{1}H NMR (400 MHz, [D$_8$]THF, −40 °C, TMS): $\delta = 7.39-7.28$ (m, 4H; Ph), 3.82 (sept, ^3J(H,H) = 6.7 Hz, 1H; NCH), 3.55 (sept, ^3J(H,H) = 6.7 Hz, 1H; NCH), 3.37 (q, ^3J(H,H) = 7.0 Hz, 4H; OEt$_2$), 1.49 (d, ^3J(H,H) = 6.8 Hz, 6H; Me), 1.12 (t, ^3J(H,H) = 7.0 Hz, 6H; OEt$_2$), 1.12 (d, ^3J(H,H) = 6.5 Hz, 6H; Me).

5: *t*BuLi (0.6 mL, 1.7 M in pentane, 1 mmol) was added to a solution of *N*,*N*-diisopropylnaphthylamide **2** (0.26 g, 1 mmol) in THF (2 mL) under nitrogen at −78 °C. The yellow suspension which formed was warmed to room temperature and dissolved at reflux on the addition of THF (1 mL). The resultant solution was stored at −30 °C for 2 d whereupon yellow crystals of **5** were deposited. Yield 25 %, m.p. 286–288 °C; elemental analysis calcd (%) for $C_{50}H_{72}Li_2N_2O_6$: C 73.98, H 8.88, N 3.45; found, C 76.90, H 7.54, N 5.63; ^{1}H NMR (400 MHz, [D$_8$]THF, −70 °C, TMS): $\delta = 8.37-7.52$ (m, 6H; Ph), 3.89 (m, 0.8H; 0.4NCH#1), 3.76 (m, 0.8H; 0.8NCH#2), 3.62 (m, 4.2H, THF + 0.2NCH#3 + 0.06NCH#4), 3.25 (m, 0.4H; 0.4NCH#5), 1.77 (m, 4H; THF), 1.64 (m, 2.4H; 0.8Me#2+0.4Me#5), 1.44 (d, ^3J(H,H) = 6.3 Hz, 1.2H; 0.8Me#2), 1.39 (d, ^3J(H,H) = 6.3 Hz, 1.2H; 0.8Me#2), 1.20 (d, ^3J(H,H) = 6.5 Hz, 1.2H; 0.4Me#5), 1.13 (d, ^3J(H,H) = 6.3 Hz, 1.2H; 0.8Me#2), 1.07 (d, ^3J(H,H) = 6.6 Hz, 0.2H; 0.06Me#4), 1.01 (d, ^3J(H,H) = 6.6 Hz, 0.2H; 0.06Me#4), 0.90 (d, ^3J(H,H) = 6.0 Hz, 0.6H; 0.2Me#3), 0.72 (d, ^3J(H,H) = 5.9 Hz, 1.2H; 0.4Me#1), 0.10 (d, ^3J(H,H) = 6.0 Hz, 0.6H; 0.2Me#3), −0.03 (d, ^3J(H,H) = 5.9 Hz, 1.2H; 0.4Me#1).

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[9] Crystal data for **4**: $C_{17}H_{28}LiNO_2$, $M = 285.34$, monoclinic, space group $P2_1/c$, $a = 9.1780(18)$, $b = 11.255(2)$, $c = 17.889(4)$ Å, $\beta = 99.55(3)^\circ$, $V = 1822.3(6)$ Å^3, $Z = 4$, $\rho_{calcd} = 1.040\ \mathrm{g\,cm^{-3}}$; Mo$_{K\alpha}$ radiation, $\lambda = 0.71073$ Å, $\mu = 0.066\ \mathrm{mm^{-1}}$, $T = 180$ K. 4427 data (4172 unique, $R_{int} = 0.0213$, $\theta < 27.48^\circ$) were collected on a Rigaku AFC5R four-circle diffractometer. The structure was solved by direct methods and refined by full-matrix least-squares on F^2 values of all data (G. M. Sheldrick, SHELXTL Manual, Version 5.1, Bruker AXS Inc., Madison, WI, **1998**) to give $wR2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2} = 0.1619$, conventional $R = 0.0594$ for F values of 2529 reflections with $F_o^2 > 2\sigma(F_o^2)$, $S = 0.997$ for 290 parameters. The Et$_2$O molecules showed positional disorder and were refined isotropically over three different sites with partial occupancies. Residual electron density max/min 0.311/−0.170 e Å^{-3}. Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-147797 **4** and CCDC-147798 **5**. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44)1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

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[14] Crystal data for **5**: $C_{50}H_{72}Li_2N_2O_6$, $M = 810.98$, triclinic, space group $P\bar{1}$, $a = 10.2370(5)$, $b = 10.5372(3)$, $c = 12.8636(5)$ Å, $\alpha = 106.429(2)$, $\beta = 95.783(2)$, $\gamma = 112.801(2)^\circ$, $V = 1191.70(8)$ Å^3, $Z = 1$, $\rho_{calcd} = 1.130\ \mathrm{g\,cm^{-3}}$; Mo$_{K\alpha}$ radiation, $\lambda = 0.71073$ Å, $\mu = 0.072\ \mathrm{mm^{-1}}$, $T = 180$ K. 7987 data (5400 unique, $R_{int} = 0.0221$, $\theta < 27.55^\circ$) were collected on a Nonius CCD diffractometer. The structure was solved by direct methods and refined by full-matrix least-squares on F^2 values of all data to give $wR2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2} = 0.2375$, conventional $R = 0.0753$ for F values of 3960 reflections with $F_o^2 > 2\sigma(F_o^2)$, $S = 1.015$ for 339 parameters. Residual electron density max/min 0.385/−0.579 e Å^{-3}.

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