5.0, 1 H), 5.09 (dd, $J$ = 8.4, 5.0, 1 H), 4.74 (d, $J$ = 6.0, 1 H), 4.42 (d, $J$ = 0.6, 1 H), 3.62 (td, $J$ = 9.0, 1.6, 1 H), 2.31 (dd, $J$ = 13.4, 6.5, 1 H), 1.68 (m, 2 H), 1.42 (d, $J$ = 6.0, 3 H), 1.29 (m, 1 H), 1.02 (t, $J$ = 8.7, 1 H); IR ($CH_2Cl_2$) 2046, 1977 cm$^{-1}$; HRMS $m/z$ 250.0288 (calcd for $C_{11}H_{14}O_2Fe$ (M − 2 CO) $m/z$ 250.0291).

**Reduction of 2b.** A solution of **2b** (235 mg, 0.77 mmol) in dry benzene (15 mL) was cooled (5 °C) under $N_2$, and a solution of diisobutylaluminum hydride in toluene (1.55 mL, 1.55 mmol) was added dropwise. After 30 min, the reaction was quenched with methanol (1 mL). The workup was the same as for **3a**. A yellow oil (**3b**) was obtained (200 mg, 85%). **3b**: $^1$H NMR ($CDCl_3$) $\delta$ 6.33 (d, $J$ = 6.0, 1 H), 5.20 (dd, $J$ = 8.7, 5.4, 1 H), 5.04 (dd, $J$ = 9.0, 5.4, 1 H), 4.72 (br d, $J$ = 6.0, 1 H), 4.42 (br s, 1 H), 3.88 (ddd, $J$ = 11.3, 6.3, 1.8, 1 H), 2.26 (dd, $J$ = 12.9, 6.6, 1 H), 1.63 (m, 2 H), 1.40 (d, $J$ = 6.3, 3 H), 1.10 (m, 1 H), 0.96 (br t, $J$ = 7.5, 1 H); IR ($CH_2Cl_2$) 2046, 1975 cm$^{-1}$; HRMS $m/z$ 250.0306 (calcd for $C_{11}H_{14}O_3Fe$ (M − 2 CO) $m/z$ 250.0291).

**Ferrier Rearrangement of 3a.** To a solution of **3a** (40 mg, 0.13 mmol) in isopropyl alcohol (5 mL) at 0 °C under $N_2$ was added *p*-toluenesulfonic acid (5 mg). The mixture was stirred at 0 °C for 8 h. The reaction mixture was poured into saturated aqueous sodium bicarbonate (1 mL). The reaction mixture was extracted with ether (3 × 10 mL), and the combined organic layers were washed with $H_2O$ (1 mL) followed by brine (1 mL). The organic phase was dried ($MgSO_4$) and concentrated to yield a yellow oil, which was purified by column chromatography ($SiO_2$) using hexanes/ethyl acetate (20:1) as eluent to give a yellow crystalline solid (34 mg, 75%). **4a**: mp 37–38 °C (hexane); $^1$H NMR ($CDCl_3$) $\delta$ 5.97 (dt, $J$ = 10.3, 4.0, 1 H), 5.67 (ddd, $J$ = 10.2, 5.0, 2.0, 1 H), 5.18 (dd, $J$ = 8.2, 5.0, 1 H), 5.10–5.07 (m, 2 H), 4.05 (sept, $J$ = 6.0, 1 H), 3.58 (td, $J$ = 8.7, 7.3, 1 H), 2.09–2.02 (m, 2 H), 1.42 (d, $J$ = 6.0, 3 H), 1.30 (d, $J$ = 6.0, 3 H), 1.23 (m, 1 H), 1.18 (d, $J$ = 6.0, 3 H), 0.85 (t, $J$ = 8.5, 1 H); IR ($CH_2Cl_2$) 2046, 1980 cm$^{-1}$; HRMS $m/z$ 348.0657 (calcd for $C_{16}H_{20}O_5Fe$ $m/z$ 348.0657).

**Ferrier rearrangement of 3b** was performed in a fashion similar to the rearrangement of **3a** to **4a**. Column chromatography ($SiO_2$) using hexanes/ethyl acetate (20:1) as eluent afforded a yellow oil (180 mg, 79%). **4b**: $^1$H NMR ($CDCl_3$) $\delta$ 5.99 (m, 1 H), 5.66 (dddd, $J$ = 10.1, 3.0, 2.8, 1.6, 1 H), 5.22 (dd, $J$ = 8.7, 5.0, 1 H), 5.08 (br s, 1 H), 5.03 (dd, $J$ = 8.8, 5.0, 1 H), 4.09 (sept, $J$ = 6.0, 1 H), 3.91 (dt, $J$ = 10.5, 5.2, 1 H), 2.10–2.03 (m, 2 H), 1.40 (d, $J$ = 6.0, 3 H), 1.26 (d, $J$ = 6.0, 3 H), 1.19 (d, $J$ = 6.0, 3 H), 1.09 (dqd, $J$ = 9.0, 5.9, 0.8, 1 H), 0.99 (ddd, $J$ = 8.9, 5.8, 0.8, 1 H); IR ($CH_2Cl_2$) 2043, 1972 cm$^{-1}$; HRMS $m/z$ 348.0648 (calcd for $C_{16}H_{20}O_5Fe$ $m/z$ 348.0657).

**Hydrolysis of Cyclic Acetal 4a.** To a solution of **4a** (210 mg, 0.60 mmol) in acetone (30 mL) was added 0.05 M $H_2SO_4$ (5 mL) under $N_2$ at room temperature. The solution was heated at reflux for 30 min. Saturated aqueous $NaHCO_3$ (10 mL) was added, and the mixture was extracted with ether (3 × 50 mL). The combined organic layers were dried ($MgSO_4$) and concentrated. Column chromatography ($SiO_2$) using hexanes/ethyl acetate (10:3) as eluent gave a yellow crystalline solid (160 mg, 87%). **5a**: mp 113–114 °C; $^1$H NMR ($CDCl_3$) $\delta$ 5.96 (m, 1 H), 5.72 (m, 1 H), 5.34 (m, 1 H), 5.22 (dd, $J$ = 8.4, 5.0, 1 H), 5.04 (ddd, $J$ = 9.1, 5.0, 0.6, 1 H), 3.64 (ddd, $J$ = 7.6, 7.6, 1 H), 2.85 (d, $J$ = 5.0, 1 H), 2.23 (m, 2 H), 1.36 (d, $J$ = 6.0, 3 H), 1.20 (m, 1 H), 0.83 (t, $J$ = 8.2, 1 H); IR ($CH_2Cl_2$) 2043, 1967 cm$^{-1}$. Anal. Calcd for $C_{13}H_{14}O_5Fe$: C, 51.00; H, 4.61. Found: C, 51.22; H, 5.11.

**Hydrolysis of Cyclic Acetal 4b.** To a solution of **4b** (160 mg, 0.46 mmol) in acetone (10 mL) was added 0.05 M $H_2SO_4$ (0.8 mL) under $N_2$ at room temperature. The solution was stirred for 16 h and worked up in a manner similar to **5a** to afford a yellow crystalline solid **5b** (95 mg, 68%). **5b**: mp 97–98 °C; $^1$H NMR ($CDCl_3$) $\delta$ 5.92 (m, 1 H), 5.71 (dddd, $J$ = 10.1, 4.0, 2.8, 1.4, 1 H), 5.41 (m, 1 H), 5.11 (dd, $J$ = 8.6, 5.0, 1 H), 4.99 (dd, $J$ = 8.9, 5.0, 1 H), 3.73 (ddd, $J$ = 9.1, 7.5, 4.9, 1 H), 2.53 (d, $J$ = 4.4, 1 H), 2.05 (m, 2 H), 1.35 (d, $J$ = 6.0, 3 H), 1.03 (dqd, $J$ = 8.8, 6.0, 0.8, 1 H), 0.92 (t, $J$ = 7.7, 1 H); IR ($CH_2Cl_2$) 2043, 1972 cm$^{-1}$. Anal. Calcd for $C_{13}H_{14}O_5Fe\cdot{}^1/_4H_2O$: C, 50.26; H, 4.70. Found: C, 50.08; H, 4.90.

**Oxidation of Unsaturated Lactol 5a.** To a solution of **5a** (60 mg, 0.2 mmol) and pyridinium dichromate (110 mg, 0.3 mmol) in $CH_2Cl_2$ (3 mL) was added freshly activated 3A molecular sieve powder (160 mg) and glacial acetic acid (1 drop). The solution was stirred until TLC showed no starting material remained (2–3 h). The mixture was extracted with ether (3 × 50 mL) and decanted. The combined organic solutions were washed successively with 0.5 M aqueous HCl (2 × 0.5 mL), saturated aqueous sodium bicarbonate solution (0.5 mL), and saturated aqueous sodium chloride (1 mL). The organic layer was dried ($MgSO_4$) and concentrated. Column chromatography ($SiO_2$) using hexanes/ethyl acetate (10:1) as eluent gave a yellow crystalline solid (46 mg, 77%). **6a**: mp 106–108 °C; $^1$H NMR ($CDCl_3$) $\delta$ 6.88 (ddd, $J$ = 9.9, 5.2, 3.5, 1 H), 6.02 (ddd, $J$ = 9.9, 2.2, 1.6, 1 H), 5.25 (ddd, $J$ = 7.9, 4.8, 0.8, 1 H), 5.17 (ddd, $J$ = 8.4, 4.8, 1.0, 1 H), 4.07 (td, $J$ = 9.4, 6.0, 1 H), 2.56 (m, 2 H), 1.44 (s, 3 H), 1.39 (m, 1 H), 0.90 (ddd, $J$ = 9.6, 8.2, 1.0, 1 H); IR ($CH_2Cl_2$) 2049, 1985, 1723 cm$^{-1}$; HRMS $m/z$ 304.0025 (calcd for $C_{13}H_{12}O_5Fe$ $m/z$ 304.0033).

**Reduction of Unsaturated Lactone 6a.** A mixture of iron pentacarbonyl (168 mg, 0.86 mmol) and 1,4-diazabicyclo[2.2.2]octane (48 mg, 0.43 mmol) in dimethylformamide/water (0.8 mL, 98:2 v/v) was flushed with $N_2$ and stirred for 5 min at room temperature. To the resulting dark red solution was added **6a** (65 mg, 0.21 mmol) in one portion. The mixture was allowed to stir at room temperature for 70 h. The mixture was treated with water (2 mL) and extracted with ether (3 × 15 mL). The combined extracts were washed with saturated aqueous sodium bicarbonate (3 × 0.5 mL) followed by saturated aqueous sodium sulfate (2 × 1 mL). The organic layer was dried ($MgSO_4$) and concentrated. The crude product was purified by column chromatography ($SiO_2$) using hexanes/ethyl acetate (20:3) as eluent to afford **7a** as a yellow oil (19 mg, 33%). **7a**: $^1$H NMR ($CDCl_3$) $\delta$ 5.28 (ddd, $J$ = 8.0, 5.0, 1.0, 1 H), 5.11 (dd, $J$ = 8.6, 5.0, 1 H), 3.95 (ddd, $J$ = 10.6, 9.4, 3.4, 1 H), 2.55 (dddd, $J$ = 17.9, 6.4, 5.0, 1.2, 1 H), 2.39 (ddd, $J$ = 17.7, 9.1, 6.8, 1 H), 2.06 (m, 1 H), 1.87 (m, 2 H), 1.65 (m, 1 H), 1.44 (d, $J$ = 6.0, 3 H), 1.36 (dqd, $J$ = 8.0, 6.2, 1.0, 1 H), 0.81 (ddd, $J$ = 9.0, 8.0, 0.9, 1 H); IR ($CH_2Cl_2$) 2046, 1967, 1737 cm$^{-1}$; HRMS $m/z$ 306.0175 (calcd for $C_{13}H_{14}O_5Fe$ $m/z$ 306.0189). The $^1$H NMR spectrum of compound **7a** was found to be identical with the spectrum of a sample generously provided by Prof. M. Franck-Neumann (Universite Louis Pasteur, Strasbourg).

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**Supplementary Material Available:** ORTEP of **2b**, and crystallographic data for **2b**, and $^{13}$C NMR spectra of compounds **3a**, **3b**, **4a**, **4b**, and **6a** (12 pages). Ordering information is given on any current masthead page.

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# Convenient Method for the Titration of Amide Base Solutions

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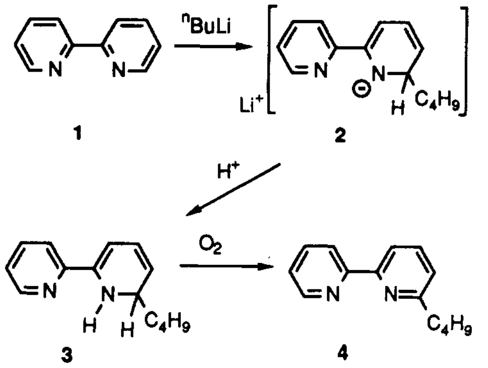
Amide bases have become of crucial importance to many procedures in organic chemistry. Several amide base solutions are available commercially, such as metal diisopropylamides and hexamethyldisilazides,[1] but due to the

**Scheme I. Formation of the Indicator**



inherent instability of the bases in solution, the titer of these commercial products often varies widely as a result of storage. In addition, direct preparations of some of these amide bases from the appropriate metal hydride and amine are heterogeneous reactions and as such are often not quantitative.[2] In many reactions, the precise knowledge of a base solution's titer is required in order to exploit properly the selectivity and characteristics of a specific base. Therefore, a method for the standardization of amide base solutions is of significant importance. None of the procedures for the titration of alkyllithium base solutions are applicable to those of amide bases, and other methods are not specific for amide bases.[3,4] A procedure that is specific for amide base concentration even in the presence of the hydroxide or alkoxide base in partially deteriorated solutions is clearly needed.

Such a procedure has been developed through the use of an indicator that is presumed to be 1,6-dihydro-6-butyl-2,2′-bipyridine (**3**), (Scheme I). The indicator was formed when an ethereal solution of 2,2′-bipyridine (**1**) was treated with 1 equiv of a 2.5 M solution of *n*-butyllithium in hexane and then the resulting deep red/blue solution was quenched with butanol. Concentration of the yellow ethereal solution afforded an air-sensitive compound that could not be completely identified, but its air-oxidation product was readily shown to be 6-butyl-2,2′-bipyridine (**4**). It was therefore reasoned that the indicator was the yellow dihydro derivative **3**, which on deprotonation generated the deep red/blue salt **2**.[5] Evaluation of this indicator and the associated color change for the titration of amide base solutions was made with consideration for the accuracy and reproducibility of the end point observed and the specificity for amide bases over alkoxide and hydroxide bases.

Two freshly prepared amide base solutions, lithium diisopropylamide (LDA) and lithium hexamethyldisilazide (LiHMDS), an aged LiHMDS[6] solution, and a commercially obtained potassium hexamethyldisilazide (KHMDS) solution were titrated repeatedly for amide base concentration against a standard solution of 2-butanol with the dihydropyridine **3** as an indicator (Table I). The results showed good reproducibility with repetition, as well as agreement with the expected concentration of the freshly prepared bases. It became clear that the rapid and distinct color change at the end point makes the dihydropyridine **3** an excellent indicator.

**Table I. Titration of Amide Base with Indicator 3**

| base | vol. of base, μL, ±4 | vol. of 1.00 M butanol, μL, ±4 | calcd [base], M |
|---|---|---|---|
| KHMDS | 200 | 102 | 0.51 |
| | 100 | 54 | 0.54 |
| | 100 | 54 | 0.54 |
| | 300 | 160 | 0.53 |
| | 200 | 108 | 0.54 |
| | | | av = 0.53 |
| LiHMDS | 100 | 101 | 1.01 |
| | 150 | 151 | 1.01 |
| | 150 | 156 | 1.04 |
| | 100 | 104 | 1.04 |
| | 200 | 206 | 1.03 |
| | | | av = 1.03 |
| LDA | 100 | 50 | 0.50 |
| | 200 | 98 | 0.49 |
| | 200 | 100 | 0.50 |
| | 250 | 126 | 0.50 |
| | 200 | 98 | 0.49 |
| | | | av = 0.50 |
| aged LiHMDS | 250 | 170 | 0.68 |
| | 200 | 140 | 0.70 |
| | 200 | 140 | 0.70 |
| | 200 | 142 | 0.71 |
| | 200 | 142 | 0.71 |
| | | | av = 0.70 |

**Table II. Titration of Total Base**

| base | vol. of base, μL, ±4 | vol. of 0.100 M CSA, mL, ±0.05 | calcd [base], M |
|---|---|---|---|
| KHMDS | 300 | 1.60 | 0.533 |
| | 300 | 1.60 | 0.533 |
| | 200 | 1.10 | 0.550 |
| | | | av = 0.54 |
| LiHMDS | 100 | 1.00 | 1.00 |
| | 100 | 1.00 | 1.00 |
| | 200 | 2.05 | 1.03 |
| | | | av = 1.01 |
| LDA | 300 | 1.50 | 0.500 |
| | 200 | 1.05 | 0.525 |
| | 300 | 1.50 | 0.500 |
| | | | av = 0.51 |
| aged LiHMDS | 500 | 4.50 | 0.900 |
| | 400 | 3.80 | 0.950 |
| | 500 | 4.60 | 0.920 |
| | | | av = 0.92 |

In order to verify the accuracy of the values in Table I, we then titrated the same bases for total base concentration against camphorsulfonic acid (CSA) with phenolphthalein as the indicator (Table II). These data confirm the accuracy and eliminate any uncertainty in the proposed stoichiometry of the reaction in Scheme I. In addition, a significant difference in the amide and total base concentration in the aged LiHMDS solution was observed, which illustrates the need for a titration that differentiates amide base from the weaker hydroxide base.

Next, the effect of hydroxide on the amide base titration was investigated. Rather than the corresponding metal hydroxide being added to the titration mixture, aliquots of the bases were partially hydrolyzed with a solution of water in THF. This allowed for a more precise and convenient method of hydroxide introduction, as exposure of

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(1) The sodium, lithium, and potassium amides can be purchased in both solid and solution form from several major supply houses.

(2) Brown, C. A. *J. Org. Chem.* **1974**, *39*, 3913.

(3) Kofron, W. G.; Baclawski, L. W. *J. Org. Chem.* **1976**, *41*, 1879.

(4) Watson, S. C.; Eastam, J. F. *J. Organomet. Chem.* **1976**, *9*, 167.

(5) The pyridine analogues of **2** and **3** have been characterized by NMR (Fraenkel, G.; Copper, J. C. *Tetrahedron Lett.* **1986**, *15*, 1825). It is significant to note that since the *n*-butyllithium adduct of pyridine itself is "deep red", a higher degree of conjugation is not necessary for absorbance in the visible region. The formation of the pyridine adduct is much slower than that of bipyridine, taking about 1 h, which makes it an inconvenient source of indicator. Under the same experimental conditions used for bipyridine, 2-phenylpyridine produces an orange dihydropyridyl anion. The fact that both pyridine and phenylpyridine produce visibly absorbing species also argues against the possibility that the indicator chromophore is an N-coordinated bidentate lithium complex.

(6) The approximately 3 month old solution in hexanes contained significant precipitates and was shaken before use.

**Table III. Titration of Amide and Total Base in Partially Hydrolyzed Aliquots**

| base | vol. of base, μL, ±4 | vol. of 1.00 M water, μL, ±4 | vol. of 1.00 M butanol μL, ±4 | total base, calcd,[a] μmol | total base, titrated,[b] μmol |
|---|---|---|---|---|---|
| LDA | 200 | 0 | 96 | 96 | 105 |
| | 200 | 0 | 100 | 100 | 100 |
| | 250 | 25 | 98 | 123 | 125 |
| | 300 | 50 | 100 | 150 | 150 |
| | 250 | 25 | 100 | 125 | 125 |
| LiHMDS | 100 | 0 | 98 | 98 | 100 |
| | 200 | 0 | 202 | 202 | 205 |
| | 150 | 50 | 102 | 152 | 155 |
| | 200 | 50 | 152 | 202 | 200 |
| | 225 | 25 | 204 | 229 | 225 |

[a] Sum of previous two values for each entry. [b] Calculated from titration as in Table II.

**Table IV. Titration of LDA in the Presence of Potassium *tert*-Butoxide**

| mass of $KO^tBu$, mg | vol. of base, μL, ±4 | vol. of 1.00 M butanol, μL, ±4 | calcd [base], M |
|---|---|---|---|
| 10 | 200 | 98 | 0.49 |
| 10 | 250 | 124 | 0.49 |
| 20 | 100 | 51 | 0.51 |
| 20 | 200 | 101 | 0.51 |
| 10 | 200 | 102 | 0.51 |
| | | | av = 0.50 |

the titration mixture to air could be avoided. The solutions were then subjected to amide base titration and then total base titration (Table III). No measurable effect on amide base titration was evident, as the difference between titrated amide base and titrated total base was precisely the amount expected from the hydrolysis. From a practical point of view, this partial quenching most accurately simulates the processes that presumably lead to decomposition during storage.

Finally, in order to evaluate the effect of alkoxides on the dihydropyridine indicator 3, potassium *tert*-butoxide was added to aliquots of the base solution before titration (Table IV). That no measurable effect was observed indicates an exceptional level of selectivity for amide bases when the dihydropyridine indicator 3 is used.

As is evident from these data, the dihydropyridine indicator 3 is accurate and specific for amide bases. It is now possible to distinguish accurately the concentration of amide base solutions even in the presence of alkoxide and hydroxide contaminants and to verify the titer of commercially prepared solutions.[7]

## Experimental Section

**General.** Ether was distilled from potasium metal/benzophenone ketyl immediately prior to use. All other materials were obtained from commercial suppliers and used without further purification unless otherwise indicated.

**Standard 1.00 M (±)-2-Butanol in Xylenes.** (±)-2-Butanol and xylenes were heated at reflux over calcium hydride for 1.5 h and then distilled. Butanol (7.412 g, 0.10 mol) was diluted to 100.00 mL with xylenes in a volumetric flask.

**Preparation of Indicator Solution from 2,2′-Bipyridine.**[8] To 3 mL of dry ether was added 5 mg of 2,2′-bipyridine. A 2.5 M solution of *n*-butyllithium in hexanes (50 μL) was added, and a deep red/blue color persisted. The 2-butanol solution was then added until the color dissipated, forming the yellow dihydropyridine indicator. This solution was stable for several hours if stored under argon (oxidizes easily).

**6-Butyl-2,2′-bipyridine (4).** Air was bubbled through a solution of the dihydropyridine for 1 h. After solvent evaporation, the residue was subjected to preparative thin-layer chromatography on silica gel with 20% ethyl acetate in hexane as the mobile phase. The major component had the following characteristics, comparable to those of the literature:[9] 300-MHz $^1H$ NMR ($CDCl_3$) δ 0.965 (t, 3 H, $J = 7.2$ Hz), 1.43 (m, 2 H), 1.78 (m, 2 H), 2.86 (t, 2 H, $J = 7.2, 7.5$ Hz), 7.15 (d, 1 H, $J = 7.8$ Hz), 7.28 (dd, 1 H, $J = 1.8, 3.9$ Hz), 7.70, (t, 1 H, $J = 7.5, 7.8$ Hz), 7.80 (dt, 1 H, $J = 1.8, 7.8$ Hz), 8.17 (d, 1 H, $J = 7.8$ Hz), 8.44 (d, 1 H, $J = 7.8$ Hz), 8.66 (d, 1 H, $J = 3.9$ Hz).

**Potassium hexamethyldisilazide (KHMDS), 15% in toluene:** purchased from Petrarch Systems, Inc., Bristol, PA.

**Camphorsulfonic Acid (CSA), 0.100 M in EtOH/$H_2O$.** Camphorsulfonic acid (1.1615 g, 0.005 mol) was diluted to 50.00 mL with 1:1 ethanol/water.

**Phenolphthalein Indicator Solution.** Phenolphthalein (5 mg) was diluted to 5 mL in 1:1 ethanol/water.

**Lithium Hexamethyldisilazide (LiHMDS), 1.00 M in Hexanes.** To a stirred solution of 10.55 mL (0.050 mol) of hexamethyldisilazane in 15 mL of hexanes at −23 °C was added dropwise 20.0 mL of 2.5 M *n*-butyllithium in hexanes. The solution was then allowed to warm and diluted to 50.00 mL with hexanes.

**Lithium Diisopropylamide (LDA), 0.500 M in Hexanes/THF.** To a solution of 7.01 mL (0.050 mol) of diisopropylamine in 20 mL of hexanes at −23 °C was added dropwise 20.0 mL of 2.5 M *n*-butyllithium in hexanes. After addition was complete, the solution was allowed to warm, and 10 mL of THF was added to dissolve the precipitate. The solution was then diluted to 100.00 mL with hexanes.

**Procedure for Amide Base Titration.** Several 10 × 75 mm glass culture tubes (oven dried) equipped with magnetic stir bars and rubber septa were charged with 2 mL of dry ether and flushed with argon. To each tube was then added 200 μL of the dihydropyridine indicator solution. The amide base solution was added dropwise until a red/blue color persisted, indicating the elimination of any background moisture. A fraction of a drop of the butanol solution was then added to regenerate the yellow color. A measured volume of amide base was added, followed by titration with the butanol solution to discharge the red/blue color. Several repetitions were performed in each tube.

**Total base titration:** same as for amide base, but performed in 1:1 ethanol/water; base titrated against standard CSA with 50 μL of phenolphthalein indicator solution (pink to colorless).

**Strong base titration in the presence of hydroxide base:** same as for strong base, but a measured volume of 1.00 M water in THF added before titration with butanol.

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(7) The purchased KHMDS was labeled as a "15% solution", which, assuming 15% mass to volume, calculates to 0.752 M. Since both the amide and total base titrations indicate the concentration to be 0.53 M, the difference must not be due to hydrolysis. It would therefore seem that the preparative reaction is not proceeding to its presumed extent, or quality control is insufficiently accurate due to a previous lack of methodology.

(8) 1,10-Phenanthroline can be used as well as 2,2′-bipyridine, but its dihydropyridine color change is less distinct.

(9) Kauffmann, T.; König, J.; Woltermann, A. *Chem. Ber.* **1976**, *109*, 3864.