with a melting point of 215–217 °C (lit. mp 210–212 °C,[1] 212–218 °C,[2] 212 °C[3]). IR (KBr, cm^{-1}): 1800, 1530, 1340, 1190, 1040, 1010. ^{1}H NMR (CDCl$_3$, δ ppm): 8.80 (d), 8.53 (d), 8.22 (t). ^{13}C NMR (CDCl$_3$, δ ppm): 129.58 (C1), 133.49, 134.86 (C4 or C6), 134.80 (C2), 139.49 (C5), 143.60 (C3), 155.71 (C7).

Registry No. I, 127472-56-4; III, 22952-25-6; sulfobenzoic anhydride, 81-08-3; saccharin, 81-07-2.

Convenient General Method for the Preparation of Primary Alkyllithiums by Lithium–Iodine Exchange

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Received March 16, 1990

The metathesis between an organic halide and an organolithium known as the lithium–halogen interchange was discovered some 50 years ago by the groups of Wittig and Gilman.[1,2] The reaction, which is a reversible process leading to an equilibrium mixture favoring the more stable organolithium,[3] has been used extensively to prepare relatively stable organometallics such as aryl-,[4–10] vinyl-,[5–9,11,12] and cyclopropyllithiums[6–9,13] by treatment of the corresponding organohalide with a more reactive alkyllithium, but the use of the interchange for the generation of an alkyllithium has, with a few notable exceptions,[14–16] met with less success.[4–9] The difficulties commonly encountered in the formation of simple alkyllithiums by lithium–halogen interchange are a consequence of the reversible nature of the reaction and the capricious behavior of alkyl halides when treated with an organolithium. Competing reactions such as β-elimination,[4–9] α-metalation,[17] and Wurtz-type coupling to produce symmetrical and mixed hydrocarbons[3,6–9] can seriously compromise the interchange as an efficient route to alkyllithiums. Recent mechanistic investigations of the lithium–halogen exchange[18–21] have revealed that many of these difficulties can be circumvented by judicious choice of experimental conditions. Herein we report a simple, convenient, and efficient method for the preparation of primary alkyllithiums by low-temperature lithium–iodine interchange that is based on the results of these mechanistic studies.

Primary alkyllithiums are readily prepared at −78 °C (dry ice/acetone bath) under an atmosphere of dry, deoxygenated argon (or nitrogen) by addition of 2.1–2.2 molar equiv of commercial *tert*-butyllithium (*t*-BuLi) in pentane to an approximately 0.1 M solution of primary alkyl iodide in dry *n*-pentane–diethyl ether (3:2 by volume). Neither the temperature nor the concentration of the alkyl iodide is critical to the success of the reaction: the interchange is exothermic, and for this reason the reaction should be run well below ambient, but the exchange proceeds rapidly and cleanly at temperatures ranging from −131 °C (N_2/pentane bath) to −23 °C (CCl_4/dry ice bath). The interchange is complete within a few min at −78 °C (or −131 °C), and the alkyllithium may be used at this temperature; however, the excess *t*-BuLi remaining in solution may complicate product isolation if an electrophile is added to the cold reaction mixture. Residual *t*-BuLi is easily removed by simply allowing the reaction mixture to stand at room temperature for ca. 1 h: the *t*-BuLi is consumed by rapid proton abstraction from diethyl ether,[22] leaving a clean solution of the less reactive primary alkyllithium. As demonstrated by the results summarized in Table I, addition of any of a variety of electrophiles to the alkyllithium solution delivers essentially pure product in good to excellent isolated yield. Significantly, the only byproduct generated by this procedure is a small quantity (typically 2–10%) of easily removed hydrocarbon derived, as detailed below, from formal reduction of the iodide during the interchange reaction.[18,23]

$$\mathrm{RCH_2I} \xrightarrow[\substack{n\text{-}\mathrm{C_5H_{12}/Et_2O} \\ -78\ ^\circ\mathrm{C},\ 5\ \mathrm{min}}]{t\text{-BuLi}} \mathrm{RCH_2Li} \xrightarrow{\mathrm{E^+}} \mathrm{RCH_2E}$$

The success of the interchange reaction depends critically on the choice of halide, alkyllithium, and solvent. Under the conditions outlined above, the mechanism of the interchange reaction between a primary alkyl iodide and *t*-BuLi most likely involves rapid, reversible attack of the alkyllithium on the iodine atom of the substrate.[18,20,24,25] Primary alkyl bromides, in contrast, react with *t*-BuLi predominantly by a process involving single-electron transfer.[18–21,26] This pronounced halogen effect on the mechanism of the interchange has a practical consequence: alkyl iodides rather than bromides should be used in the exchange reaction for the preparation of primary alkyl-

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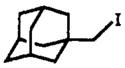
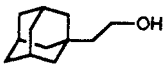
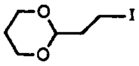
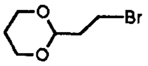
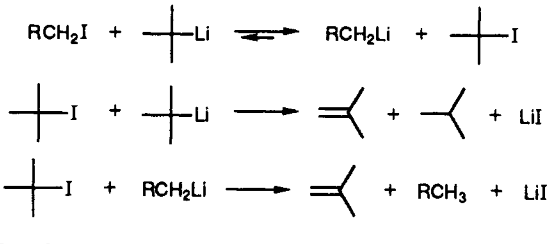
Table I. Preparation of Primary Alkyllithiums by Lithium–Iodine Interchange[a]

entry	iodide	E^{+} [b]	product	yield,[c] %
1	$CH_3(CH_2)_3I$	$CH_3(CH_2)_3CHO$	$CH_3(CH_2)_3CH(OH)(CH_2)_3CH_3$	91
2	$CH_3(CH_2)_7I$	$(CH_3)_2C{=}O$	$CH_3(CH_2)_7C(OH)(CH_3)_2$	93
3	$CH_3(CH_2)_{16}CH_2I$	H_2O	$CH_3(CH_2)_{16}CH_3$	96
4	$(CH_3)_3CCH_2I$	$CH_3(CH_2)_2CHO$	$(CH_3)_3CCH_2CH(OH)(CH_2)_2CH_3$	89
5	c-$C_5H_9CH_2I$	CO_2	c-$C_5H_9CH_2CO_2H$	91
6	$Ph(CH_2)_2I$	PhCHO	$Ph(CH_2)_2CH(OH)Ph$	91
7		$(CH_2O)_n$		83
8		$BrCH_2CH_2Br$		92

[a] The alkyllithium was generated at −78 °C by addition of 2.1–2.2 molar equiv of *t*-BuLi in *n*-pentane to a 0.1 M solution of the iodide in *n*-pentane–diethyl ether (3:2 by volume). The cooling bath was removed 5 min after complete addition of *t*-BuLi, and the reaction mixture was allowed to stand at room temperature for 1 h. [b] The reaction mixture was recooled to −78 °C and an excess (typically 2 equiv) of electrophile was added. [c] Isolated yield of chromatographically pure product.

lithiums. In this connection, it is worth noting explicitly that the interchange procedure described above works well only for primary iodides: nonbridgehead tertiary alkyl iodides undergo rapid dehydrohalogenation when treated with *t*-BuLi[7–10] and, due to the mechanistic complexity of the lithium–halogen exchange between a secondary alkyl halide and *t*-BuLi,[18,21,23] it has not yet proved possible to devise conditions for the clean production of secondary alkyllithiums by the interchange reaction.[18,23]

The rationale for the use of *t*-BuLi in the interchange, which was first advocated more than a decade ago by the groups of Corey[27] and Seebach,[11] follows from two considerations: (1) the exchange equilibrium is favorable when a primary iodide is treated with a tertiary organolithium[3] and (2) the interchange is effectively rendered irreversible by the use of 2 molar equiv of *t*-BuLi since an equivalent of *t*-BuLi consumes the *tert*-butyl iodide generated in the interchange to give isobutane, isobutylene, and lithium iodide.[11,27,28] A further, and unfortunately unavoidable, consequence of the use of *t*-BuLi in the interchange protocol is the formation of a small but nonnegligible quantity of hydrocarbon formally derived from reduction of the alkyl iodide. As shown in the diagram below, this side reaction is the result of rapid reaction of the product RCH_2Li with the cogenerated *t*-BuI to give RCH_3, isobutylene, and LiI.[23] When the hydrocarbon is the desired product, this formal reduction is of no consequence and addition of water to the organolithium solution affords a virtually quantitative yield of material (Table I, entry 3). In those instances where the organolithium is trapped by an electrophile other than a proton, formation of the easily removed hydrocarbon accounts for 2–10% of the material balance.



The choice of solvent for use in the lithium–iodine interchange is of central importance to the success of the reaction. The solvent system used in the general procedure described above for the production of alkyllithiums (n-C_5H_{12}–Et_2O; 3:2 by vol) is that found by Applequist and O'Brien, in their classical study of exchange equilibria,[3] to give clean lithium–halogen interchange. While the proportions of the mixture are not crucial to the success of the interchange, it is necessary to run the reaction in a solvent system that contains diethyl ether. Thus, although we have successfully conducted interchange reactions in pure diethyl ether as well as in solutions of pentane containing as little as 6 molar equiv of Et_2O per mole of *t*-BuLi,[29] the reaction proceeds very slowly (if at all) in pure *n*-pentane.[18,21,29] Moreover, the use of THF (or other strongly coordinating Lewis bases such as TMEDA) is detrimental to the success of the interchange reaction of a primary alkyl iodide with *t*-BuLi and should be avoided. Indeed, exploratory studies have demonstrated that β-elimination and Wurtz-type coupling are the predominant modes of reaction of *t*-BuLi with primary alkyl halides in THF solution.[29] The etiology of these rather dramatic solvent effects on the course of reactions of *t*-BuLi with alkyl halides remains obscure but it is most probably related to the degree of association of *t*-BuLi in the various solvents.[2] Recent investigations have revealed that *t*-BuLi is predominantly tetrameric in hydrocarbon solution,[30] dimeric in diethyl ether,[22] and monomeric in THF.[31] While it is tempting to speculate that ether-solvated dimeric *t*-BuLi [viz. $(t\text{-BuLi}{\cdot}2OEt_2)_2$][22] is responsible for the chemistry described above, there is as yet no direct evidence that the lithium–iodide interchange is mediated by dimeric *t*-BuLi.

In summary, lithium–iodide interchange between *t*-BuLi and a primary alkyl iodide provides an experimentally simple, efficient, and clean method for the preparation of primary alkyllithiums. A noteworthy feature of the procedure described above is the production of solutions of RCH_2Li that are free from contamination by residual *t*-BuLi or coupling products.

Experimental Section

Proton and carbon-13 magnetic resonance spectra were recorded as solutions in $CDCl_3$ on an IBM AF-270 NMR and shifts are referenced with respect to internal Me_4Si. A Perkin-Elmer Model 283 instrument was used to record infrared spectra. The purity of starting materials and products was assessed by analytical gas–liquid chromatography (GC) using either a Hewlett-Packard

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Model 5890 chromatograph equipped with a flame-ionization detector and a 25 m × 0.20 mm cross-linked methyl silicone (0.33-μm film thickness) fused-silica capillary column or a Hewlett-Packard 5870B GC/MSD system with Chemstation software operating at 70 eV and fitted with a 12.5 m × 0.20 mm cross-linked dimethyl silicone fused-silica capillary column.

All reactions involving alkyllithiums were performed in glassware that had been flame-dried under an atmosphere of dry argon, and all manipulations of organolithiums were conducted by using standard syringe/cannula techniques[32] under an atmosphere of dry, oxygen-free argon that had been passed through a 5 cm × 50 cm glass column containing an activated BASF R3-11 copper catalyst. Diethyl ether was freshly distilled from dark-purple solutions of sodium/benzophenone. Dry, olefin-free *n*-pentane was obtained by repeated washings of commercial *n*-pentane with concentrated sulfuric acid until the acid layer remained clear, followed by washing successively with several portions of water, saturated aqueous sodium bicarbonate, and water, drying ($MgSO_4$), and distillation of the purified pentane under nitrogen from lithium aluminum hydride. Acetone (Baker, analytical grade) was dried over calcium sulfate and distilled. Sodium iodide was dried at 100 °C (ca. 5 mm) for 8–10 h in a vacuum oven. The concentration of commercial solutions of *t*-BuLi in *n*-pentane (Aldrich) was determined immediately prior to use by titration with *sec*-butyl alcohol in xylene using 1,10-phenanthroline as indicator.[33] Thin-layer chromatography (TLC) was performed on E. Merck precoated (0.2-mm) silica gel 60 F_{254} plates: visualization was accomplished by spraying with 10% ethanolic phosphomolybdic acid and heating. Reaction products were purified by flash chromatography, conducted as described by Still,[34] using Universal Scientific 32–60 μm silica gel.

Neopentyl iodide and *n*-butyl iodide were purchased from Aldrich and the remaining iodides were prepared by iodine exchange (NaI in acetone) from commercially available bromides or, via the mesylate, from alcohols. The physical and spectroscopic properties of the iodides were fully in accord with those reported for 1-iodooctane,[35] 1-iodooctadecane,[36] (iodomethyl)cyclopentane,[37] phenethyl iodide,[38] 1-(iodomethyl)adamantane,[39] and 2-(2-iodoethyl)-1,3-dioxane.[40] Prior to use, the alkyl iodides were purified by passage through a short column of silica gel, using *n*-pentane as eluent.

General Procedure for the Preparation of Primary Alkyllithiums. An appropriately sized round-bottomed flask, fitted with Teflon-coated magnetic stirring bar and rubber septa, was flushed with a steady stream of dry, oxygen-free argon (introduced through the septa using 22-gauge stainless steel needles) and the entire assembly was flame-dried using a Bunsen burner and then allowed to cool to room temperature while being swept with argon. The cool, dry flask was charged with the primary alkyl iodide and enough dry *n*-pentane–diethyl ether (3:2 by volume) to give an approximately 0.1 M solution of the iodide. All additions were performed by using argon-flushed syringes and a positive pressure of argon was maintained within the flask during all subsequent operations. The solution was cooled to −78 °C by lowering the flask into a dry ice–acetone bath, the stirrer was started, and 2.1 to 2.2 molar equiv of a solution of *t*-BuLi in *n*-pentane was then added dropwise via an argon-flushed syringe. Stirring was continued at −78 °C for an additional 5 min following the addition, the cooling bath was then removed, and the mixture was allowed to warm and stand at room temperature for 1 h to consume unreacted *t*-BuLi. Argon flow was adjusted to a minimum to avoid loss of solvent by evaporation at room temperature. The mixture was then re-cooled to −78 °C, an excess of the electrophile (typically 2.0–2.2 molar equiv) was then added (in the case of 1,2-dibromoethane and carbon dioxide, the alkyllithium solution was added to the electrophile in diethyl ether), the cooling bath was removed, and the reaction mixture was worked up in the usual manner.[9]

The products listed in Table I are known compounds whose physical and spectroscopic properties were fully in accord with those reported for 5-nonanol,[41] 2-methyl-2-decanol,[42] *n*-octadecane,[43] 2,2-dimethyl-4-heptanol,[44] cyclopentylacetic acid,[45] 1,3-diphenyl-1-propanol,[46] and 2-(2-bromoethyl)-1,3-dioxane.[47]

Acknowledgment. This work was supported by a generous grant from the Humphrey Chemical Company, North Haven, CT, and by the Connecticut Department of Higher Education under Grant No. 90-630. We thank Professor Ei-ichi Negishi of Purdue University for a helpful exchange of information prior to publication of a related study of the lithium–iodine exchange by his group.

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Clean and Convenient Procedure for Converting Primary Alkyl Iodides and α,ω-Diiodoalkanes into the Corresponding Alkyllithium Derivatives by Treatment with *tert*-Butyllithium

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Whereas lithium–halogen exchange[2] has been widely used for generating alkenyl- and aryllithiums,[3] its application to the preparation of alkyllithiums has not been widely reported. We have recently noted that the reaction of alkyl iodides with 2 equiv of *t*-BuLi can provide a clean, high-yielding, and convenient method for preparing the corresponding alkyllithiums as discrete products.[4] This method has also been used to generate alkene- and alkyne-containing alkyllithiums for both mechanistic[5] and synthetic[6] purposes. These results suggested to us that the method might provide a general route to primary alkyllithiums, which in some cases is superior to the previously developed alternatives.[7] We now report that this

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