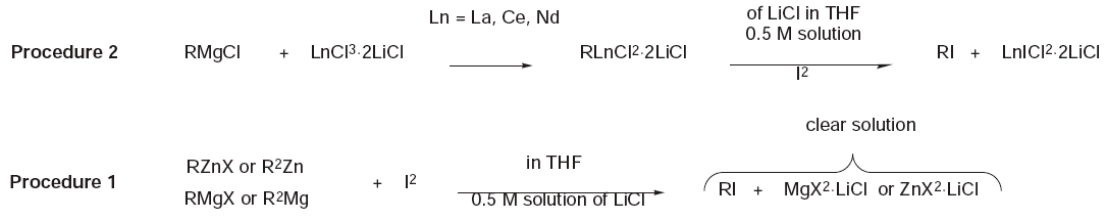
K 481



Titration of organomagnesium, organozinc, and organolanthanide compounds.

Saturated Solution of LiCl in THF
Anhydrous LiCl (100 mmol) was placed in an argon-flushed flask and dried under high vacuum (1 — 10^{-2} mbar) for 4 h at 140 °C. After cooling to r.t., anhydrous THF (200 mL) was added and the mixture was stirred for 24 h at r.t. until the LiCl was completely dissolved, resulting in the formation of a 0.5 M solution of LiCl.

Titration Using I2
A 10-mL round-bottom flask equipped with a magnetic stirring bar and a septum was heated with a heat gun under reduced pressure and cooled to r.t. under an argon atmosphere. The dry flask was charged with accurately weighed I2 (254 mg, 1 mmol), fitted with a rubber septum, and flushed with argon. The sat. solution of LiCl in THF (3–5 mL) was added and stirring was started. After the iodine was completely dissolved, the resulting brown solution was cooled to 0 °C in an ice bath and the organometallic reagent was added dropwise via a 1.00-mL syringe (0.01-mL graduations) until the brown color disappeared. The amount consumed contains 1 equiv of the organometallic reagent relative to iodine in the case of monoorganometallic reagents and 0.5 equiv for diorganometallic reagents.

It is possible to determine the concentration of most organolithium compounds using our procedure with the exception of some secondary and tertiary alkyl lithiums in which a facile elimination of HI from the alkyl iodide formed during titration is possible (for example *s*-BuLi and *t*-BuLi).

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