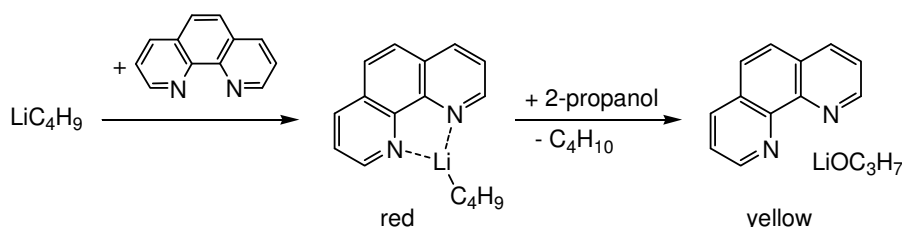


## Titration Solution for Quantitative Analysis of BuLi 2-Propanol 1.0 M in toluene with 0.2% of 1,10-phenanthroline

**Product Aldrich #689327**

### Application

This ready-to-use reagent is for simple determination of BuLi-activity in hydrocarbons. It is designed to analyze product Fluka #20160 or product Aldrich #186171 (both 1.6 M BuLi in hexanes) with an accuracy of up to +/- 3%. The titration's end is shown by a color change of the incorporated indicator 1,10-phenanthroline (see Scheme below). Each stock bottle of 5 mL provides a sufficient volume for one double estimation of 1.6 M BuLi solution.



### Composition

1000 mL of *titration solution for quantitative analysis of BuLi (product Aldrich #689327)* contain:

- 60.1 g of dry 2-propanol (active ingredient for BuLi neutralization, corresponds to  $c = 1.0 \text{ M}$ )
- 2.0 g of 1,10-phenanthroline (indicator)
- in dry toluene

### Required Materials

- Min. 4 disposable syringes, volume 1 mL, graduation 0.01 mL (recommended product Aldrich #Z230723)
- Min. 4 disposable needles (recommended product Aldrich #Z192570)
- Min. 20 mL of inert dry solvent e.g. toluene (recommended product Fluka #89677)
- 50 mL three-necked flask (preferred material glass) with matching septa
- Magnetic stirrer and stirring bar
- Ice bath (to realize  $0 - 4 \text{ }^\circ\text{C}$ )
- Protective gas supply (Argon preferred) with pressure balance valve



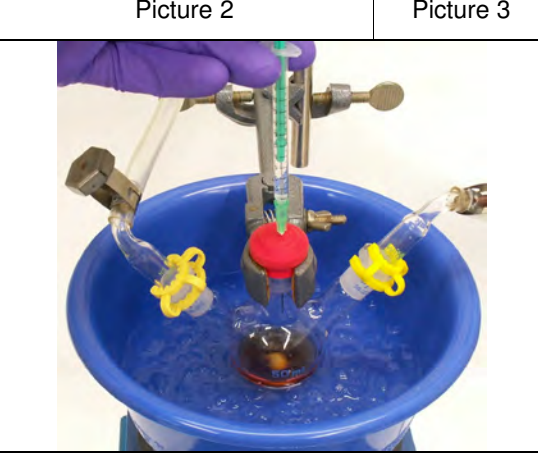
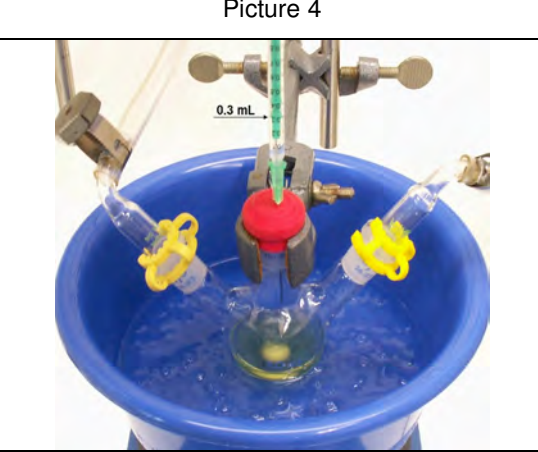
### Procedure

- Connect the three-necked flask with the protective gas supply. Insert stirring bar, close middle flask-opening with the septum and flush set-up thoroughly with protective gas for 1-2 min (Picture 1).
- *Note: Do not over-pressurize the closed flask!*
- Minimize gas stream.
- Cool down the set-up to ca.  $+4 \text{ }^\circ\text{C}$  using an ice bath.



Picture 1

## Procedure

<ul style="list-style-type: none"> <li>Inject ca. 10 mL of dry inert solvent (e.g. toluene) followed by <b>exactly</b> 1.0 mL of the BuLi sample solution to be tested (Picture 2).</li> <li>Load the 1 mL syringe with <b>exactly</b> 1.0 mL of <i>titration solution Aldrich #689327</i> (Picture 3). Dispose exceeding amounts (&gt; 1.0 mL) of <i>titration solution Aldrich #689327</i>.</li> <li><i>Note: The liquid inside the syringe must be free of bubbles.</i></li> </ul>		
<ul style="list-style-type: none"> <li>Inject the first 1.0 mL of <i>titration solution Aldrich #689327</i> slowly to the stirred sample solution (Picture 4). Its color will immediately turn to dark red (simultaneously the desired BuLi neutralization by 2-propanol starts).</li> <li>Load the 1 mL syringe with another 1.0 mL (<b>exactly</b>) of <i>titration solution Aldrich #689327</i> from the stock bottle (Picture 3).</li> </ul>		
<ul style="list-style-type: none"> <li>Slowly inject the 2<sup>nd</sup> charge of <i>titration solution Aldrich #689327</i> drop by drop to the stirred sample solution. The color change dark red / yellow indicates the neutralization of all active BuLi in the sample (Picture 5).</li> <li>At the neutralization point in the given example (Picture 5) the piston inside the syringe stopped at scale 0.3 mL. The totally consumed volume of 1.0 mL + 0.7 mL = 1.7 mL represents an active BuLi concentration of 1.7 mol/L.</li> </ul>		

For verification the above mentioned procedure should be repeated from the same bottle of *titration solution Aldrich #689327*. Please note that the stock bottle's membrane (see Picture 3) does not guarantee complete air tightness once perforated. Follow-up titration experiments should be carried out as soon as possible. Opened bottles are inappropriate for long-time storage.