

## Organometallic Reaction – Sub-Ambient Temperatures & Inert Atmosphere

Synthesis of 5-Phenylnonan-5-ol

### ■ MiniBlock XT

#### Summary

The synthesis of 5-phenylnonan-5-ol was carried out at sub-ambient conditions in a dry inert atmosphere due to the sensitivity of the reaction to moisture and oxygen. Despite the difficult reaction conditions, reproducible results and yields were obtained with the MiniBlock XT.

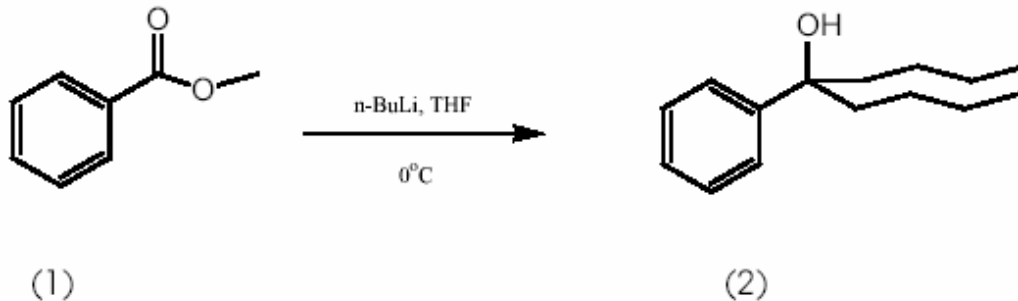
#### XT Background

The MiniBlock XT was developed to enable solution phase parallel synthesis in a variety of laboratory contexts. The XT provides both a familiar synthesis system and an expandable format for new needs as laboratory processes evolve. Its features range from traditional (e.g. operational ease, spatial efficiency, powerful mixing, reaction visibility) to sophisticated automation integration (e.g. parallel evaporators, liquid handlers, work-up including Mettler-Toledo's ALLEX liquid – liquid extraction workstation).



#### Reaction Introduction

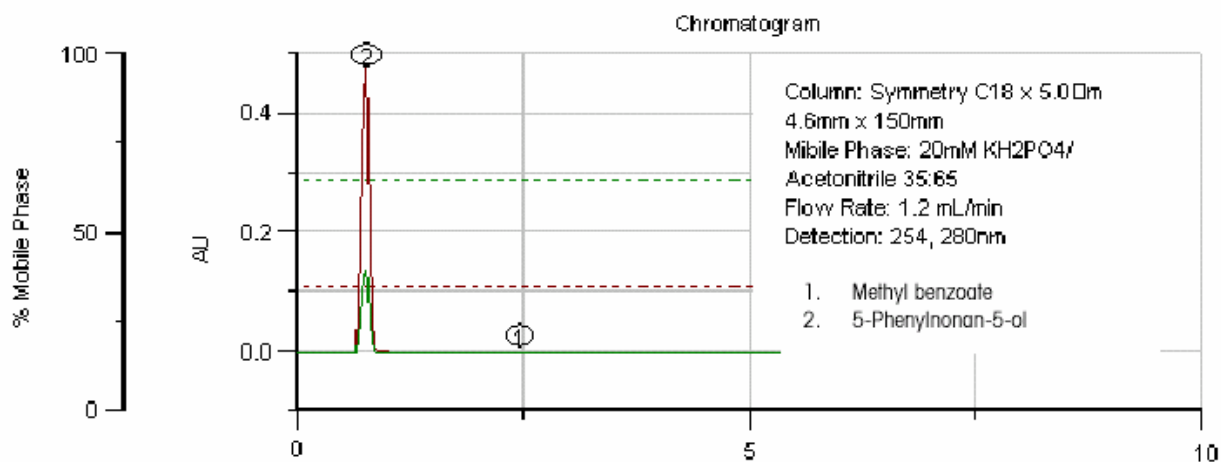
Organometallic reactions provide synthetically useful applications in chemistry in the synthesis of novel compounds. The synthesis of 5-phenylnonan-5-ol (2) below demonstrates the reaction between butyllithium and methyl benzoate (1) in the Mettler-Toledo AutoChem MiniBlock XT.



### Procedure: Preparation of 5-Phenylnonan-5-ol

A 6-reaction vessel solution phase MiniBlock equipped with crossed-shaped magnetic stir bars and the inert-gas manifold was charged with 20mls of anhydrous THF. The vessels were evacuated and the solvent degassed by continuously purging with dry nitrogen and evacuating for 30 minutes. Benzoic acid methyl ester (1.5 mmoles) was added to each vessel and the MiniBlock was cooled to  $0^\circ\text{C}$  in an ice bath. 2.5M N-butyllithium (1.4ml) was added dropwise with a syringe. When the addition was completed, the reaction was left stirring for an hour, the cooling bath was removed and the block was left to warm to room temperature. Saturated aqueous ammonium chloride was used to quench the reaction and ethyl acetate was added.

The content of each reaction vessel was transferred to a separatory funnel and the organic layer separated. The aqueous layer was washed 3 times with EtOAc. All the organic layers were combined and dried over anhydrous magnesium sulfate. Filtration and concentration by rotary evaporation yielded an off-white solid.



## Results And Discussion

The reactions gave crude yields of (97.5 – 98.4%) and HPLC analysis confirmed 98.8– 100% purity of the desired product, 5-phenylnonan-5-ol (2).

## Conclusion

This organometallic reaction is an example of sensitive synthesis which can be performed on the XT to yield high quality results - comparable to results using traditional laboratory apparatus. With the XT, the Chemist can benefit from the improved productivity of parallel synthesis while using familiar techniques and minimal bench space.