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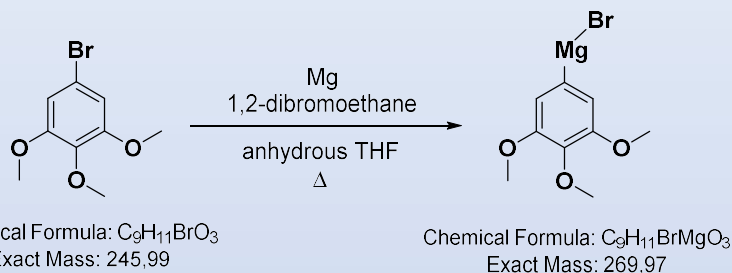
MS spectroscopy: a powerful tool for organometallic intermediate identification ?

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Introduction

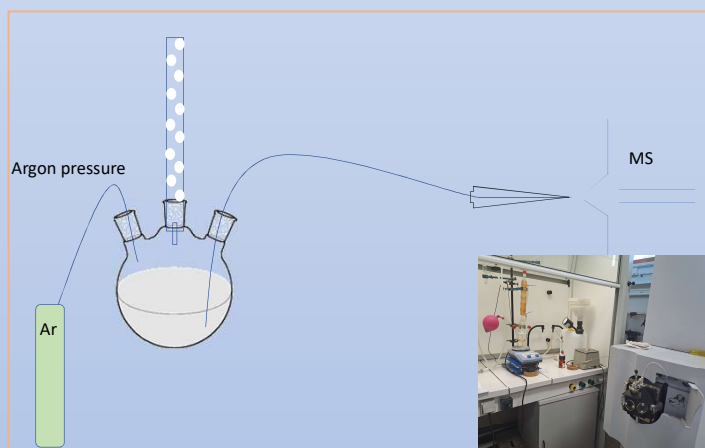
Our research are interested in the preparation, reactivity and use of organometallic reagents in organic synthesis. **Because these reagents generally require drastic conditions, in particular anhydrous conditions and temperature control to guarantee their stability**, identifying these key intermediates remains a major challenge for understanding the reaction mechanisms involved.

Because Mass Spectrometry is a powerful method for identifying complex species, including trace amounts in a medium, we focus our attention on the development of a MS method for the characterization of organometallic intermediates. The main advantage of MS for this type of challenge is that analysis and acquisition times is shorter than with NMR, commonly used to identify organometallic intermediates. MS also allows the reaction medium to be analyzed in conventional solvent, and does not require expensive deuterated solvents. However, several difficulties may be encountered during this study, mainly due to the sensitivity of organometallic derivatives to temperature (problem for the ionization step) and humidity (need to work under an inert atmosphere).



Organomagnesium derivative (RMgX, Grignard reagents) are chosen as models for the preliminary study to explore the feasibility of such a MS characterization method. The results and difficulties encountered during this study will be presented and discussed.

How to work under inert condition ? Without dioxygen and moisture



In the reactor

Mg: 6.0 mmol
anhydrous THF < 10 ppm H_2O (Karl-Fisher): 0.8 mL
1,2-dibromoethane: 3 drops
heat

Add

1-bromo-3,4,5-trimethoxybenzene: 4.0 mmol in 9 mL anhydrous THF

Sample transfert

Ar (alphagaz 1): 0.5-1.0 bar
microTofQ (Bruker) equipped with ESI
flushing with anhydrous THF during 30 min

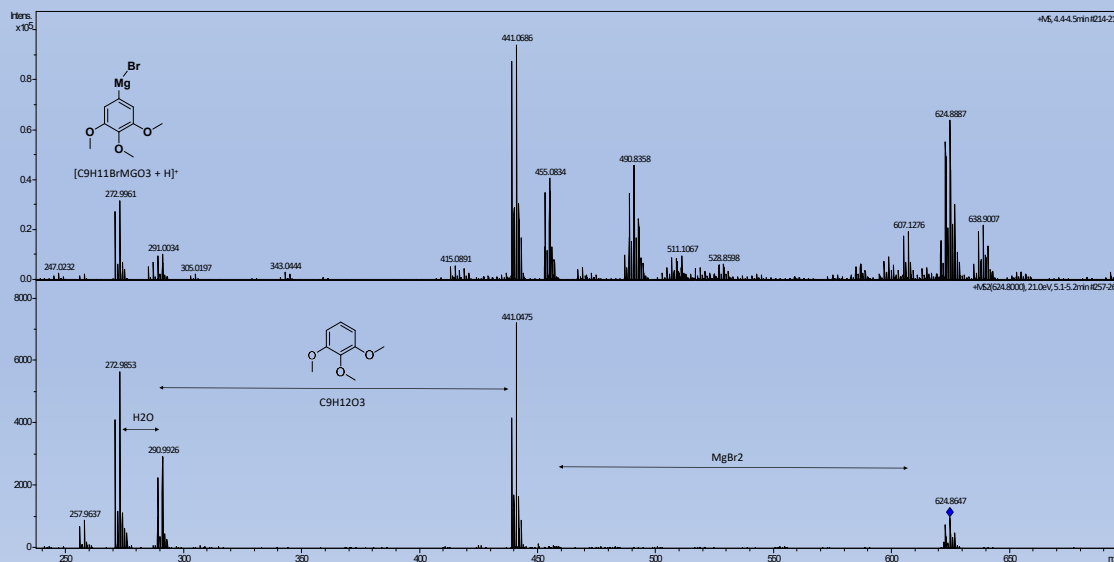
Results

Source (positive mode)

- Capillary: 4500V
- Nebulizer: 0.5 bar
- Dry gaz: 5 l/mn
- Dry temperature: 120 °C

Collision cell

- Ar (alphagaz 1)
- MS mode 7 eV
- MSMS mode 20 eV



Conclusion

We only identify a few complexes, such an initial result encourage us to carry on with our study. Particularly, we will improve the experimental setup to minimize the risk of hydrolyze of the organometallic reagents and enhance an efficient transfer of the sample. The aim of our investigations is to propose a convenient and easy-to-use method to improve knowledges about organometallic reagents (e.g. RMgX, R'Li, ...).