CONVERSION OF METAL BOROHYDRIDES IN REACTION WITH BH_3

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Boron-hydrogen based materials have attracted a lot of interest since their discovery a hundred years ago. Metal dodecaborates ($M_xB_{12}H_{12}$) are a versatile class of compounds which have shown its potential application in cancer treatment, polymer chemistry and as ionic conductors, they are commercially available but at a very high price. The synthesis of $M_xB_{12}H_{12}$ on sizable scale was a great effort of many groups of researchers [1].

In this study, we proposed a new and facile route for the synthesis of $M_xB_{12}H_{12}$ (M=Li, Na, K, Mg, Ca) from borane complex ($H_3B{\cdot}L$, L= Lewis base) and borohydrides in diglyme using an autoclave. The enclosed high-pressure system provided by autoclave can not only increase the solubility of low-solubility borohydrides (such as K, Mg, Ca) in the solvent but also keep all active gaseous product formed inside. For high solubility borohydrides (such as Li, Na), an ambient pressure apparatus (Schlenk line) was used for synthesis, giving a great benefit to the investigation of the reaction mechanism by solution NMR. The as-synthesized samples were further measured by means of numerous techniques (PXRD, SR-XPD, FTIR, NMR, TGA/DSC, ICP). From the ex-situ ^{11}B NMR study, the mechanism of $B_{12}H_{12}^{2-}$ formation from BH_4^- was elucidated, $B_3H_8^-$ and $B_{11}H_{13}^{2-}$ were found as main intermediates. We have developed a facile synthetic method to obtain high purity $M_xB_{12}H_{12}$ (M=Li, Na, K, Mg, Ca) and high in yields by directly reacting the corresponding MBH_4 salts with $DMS{\bullet}BH_3$. The new synthetic method paves the way to large scale $M_xB_{12}H_{12}$ synthesis and thus to their wider applications.

References

[1] Hansen, B. R. S.; Paskevicius, M.; Li, H.-W.; Akiba, E.; Jensen, T. R. Metal boranes: Progress and applications. Coord. Chem. Rev. 2016, 323, 60−70.

Jian Wang was born in 1992 in Hubei, China. Currently, he is a PhD student in the research group of Prof. Yaroslav Filinchuk at Université catholique de Louvain, Belgium. He is working on the synthesis of boron clusters and CO_2 reduction by borohydrides. He has studied borohydrides containing both alkali (alkali earth) and alkylammonium borohydrides for high purity and high yield $M_xB_{12}H_{12}$ production.