Alkali Bis(trifluoromethylsulfonyl)imide Molten Salts as New Solvents for Catalysis

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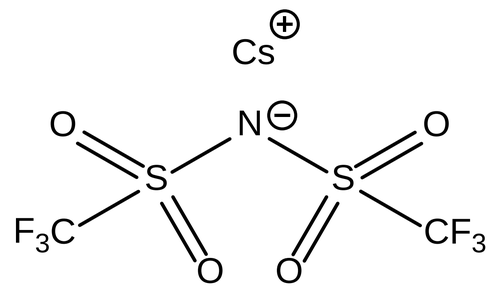
Low Temperature Molten Salts

Ionic Liquid
- Organic cation and anion
- T_m < 100 °C
- T_{stable} < 250 °C [1]

Low temperature molten salts
- Inorganic cation + organic anion
- Salt mixtures
- 100 °C < T_m < 250 °C
- T_{stable} > 250 °C

High temperature molten salts
- Inorganic cation + anion
- T_m > 400 °C
- T_{stable} >> 400 °C

- Bridge the gap between ionic liquids and classical high temperature molten salts
- Novel class of reaction media for reactions at temperatures between 150 °C and 350 °C
- Enhanced reactivity
- Tuneable selectivity
- Basic conditions possible
- Catalyst stabilization



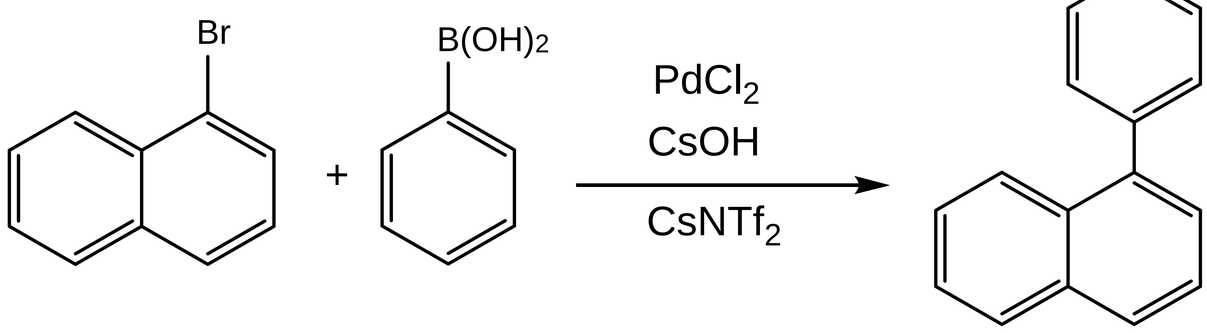
Cesium Bis(trifluoromethyl-sulfonyl)imide

$CsNTf_2$

T_m / °C	120
T_{decomp} / °C	462
$\rho_{200\ °C}$ / g cm^{-3}	2.21
$\eta_{200\ °C}$ / mPas	15

Experimental Setup

Test reaction: Suzuki cross-coupling

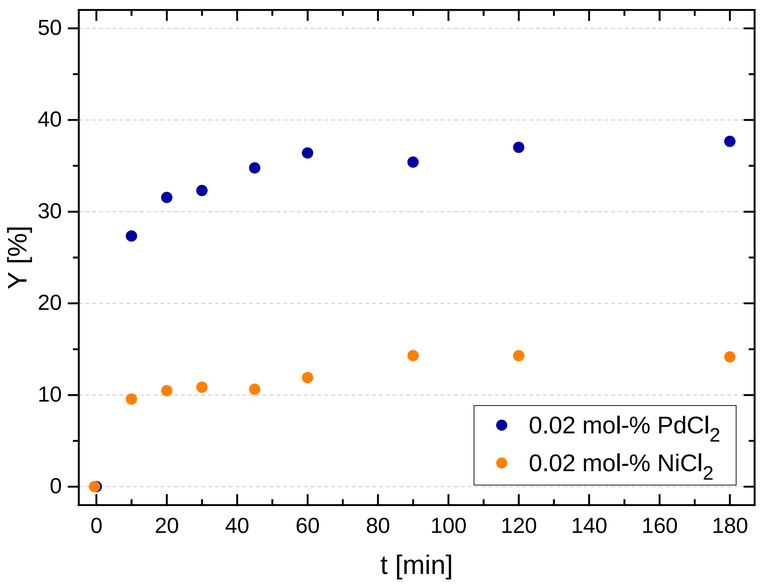


Reaction conditions:
- $CsNTf_2$ as solvent (2 eq.)
- CsOH as base (1 eq.)
- Inert gas: Argon, atmospheric pressure
- 180 °C
- Batch experiments in a glass flask with magnetic stirring bar
- Sample analysis with offline-GC

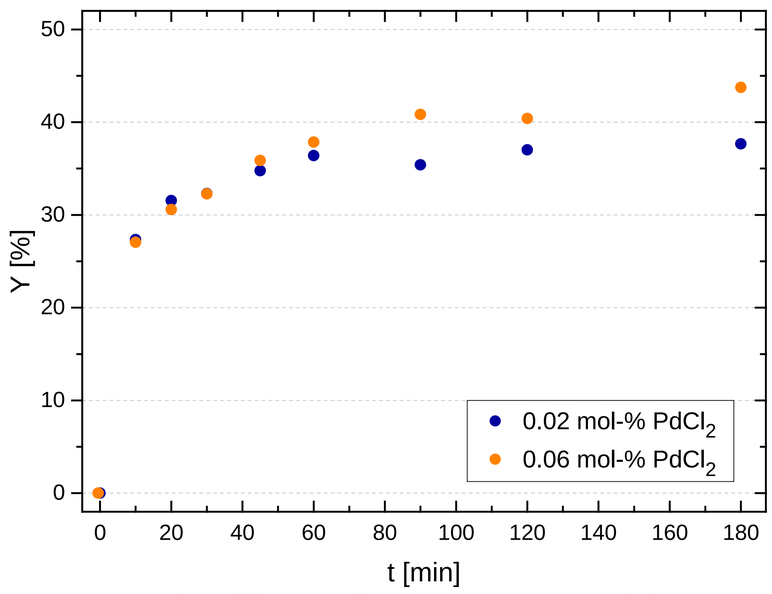
Catalyst System:
- $PdCl_2$ (0.02 mol-%) without further treatment
- Liquid-liquid biphasic system with the catalyst in the molten salt phase

Variations:
- Phenylboronic acid derivates
- Catalyst or Catalyst amount

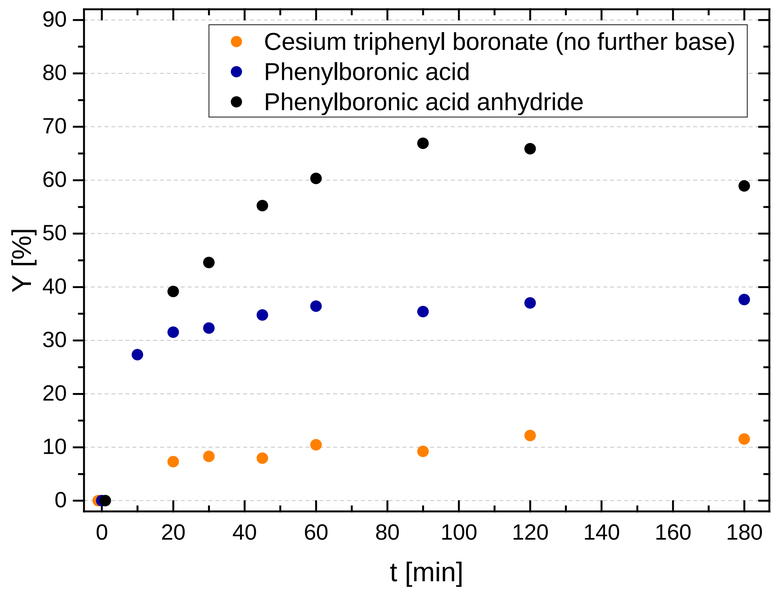
First Results



Moderate Yields with ligandless Pd-catalyst; even with $NiCl_2$



No significant improvment with more catalyst



Best results with Phenylboronic anhydride

Outlook

- Further experiments with Phenylboronic anhydride
- Temperature variations
- Optimization of the experimental setup (e.g. enlarge of interface area by better stirring)
- Variations of $CsNTf_2$ amount

[1] S. Werner, M. Haumann, P. Wasserscheid, Annual Review of Chemical and Biomaolecular Engineerng 1 (2010) 203-230.

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