7. Experimental Part

7.1 General

All commercially available substances were purchased from Aldrich, Merck, or Acros, and used without further purification. Solvents were purified and dried – if necessary – according to standard methods.[183] The catalyst $Pd(PPh_3)_4$ was synthesized according to literature[184] and stored in the dry-box. For reactions with this catalyst, the solvents, solutions or suspensions were degassed by a repeated cycle of evacuation and flushing with nitrogen. The nitrogen was purchased from Messer-Griesheim as Nitrogen 4.0 or 5.0.

7.1.1 Analyses

NMR spectrometry:

The spectra were recorded on a Bruker WH 270 MHz or AC 500 MHz with the solvent itself as inner standard. If not stated otherwise, measurements were done at room temperature. All shifts are given in ppm. The deuterated solvents were purchased from Merck or Deutero GmbH.

EI and FAB(+) mass spectrometry:

Spectra were recorded with a Varian MAT 771 or MAT 112 S spectrometer.

MALDI-TOF mass spectrometry:

Spectra were recorded with a Kratos MALDI 3 from Shimadzu. Please note that values given for signals in MALDI generally refer to the lowest mass isotope of a specific ion (not for the isotope with highest intensity!).

Elemental analysis:

It was used a Perkin-Elmer EA 240.

Melting points:

They were recorded using Büchi 510 (open capillaries, uncorrected values).

7.1.2 Chromatographic Methods

Analytical TLC:

Reactions were checked by TLC with TLC-ready charts by Merck (no. 5554, aluminium sheets with silica gel Si 60 with fluorescence indicator F_{254}), or TLC-ready charts by Macherey-Nagel (Polygram Alox N/$UV_{254,}$ ready foils with 0.2 mm aluminium oxide with

fluorescent indicator F_{254}). Detection was in UV light with wavelength λ = 254 or λ = 366 nm. If not stated otherwise, silica gel TLC charts were used.

Preparative column chromatography:

The chromatography was run with Merck flash silica gel (230-400 mesh ASTM, grain size 40-60 pm), Machery-Nagel silica gel 60 M (230-400 mesh ASTM, grain size 40-63 pm), or Fluka aluminium oxide neutral (Typ 507 C, 0.05-0.15 mm)

Analytical GPC:

Measurements were performed with a Waters Assoc. 150-c Alc/GPC chromatograph by using the column set Waters Styragel HR columns. As mobile phase, THF was used. Detection was by a Waters 410 RI detector or a 484 UV/VIS detector against polystyrene as calibration standard.

Preparative GPC:

Separation was by using a Waters machine with UV detection; the mobile phase was THF. Separation columns were Waters Styragel HR columns. In some cases, material was contaminated with THF oligomers after preparative GPC;[185] the material could be purified by precipitation as described in the corresponding procedures.