Electronic Supplementary Material (ESI) for Reaction Chemistry & Engineering.

Electronic Supplementary Information

Design and 3D Printing of a Stainless Steel Reactor for Continuous Difluoromethylations Using Fluoroform

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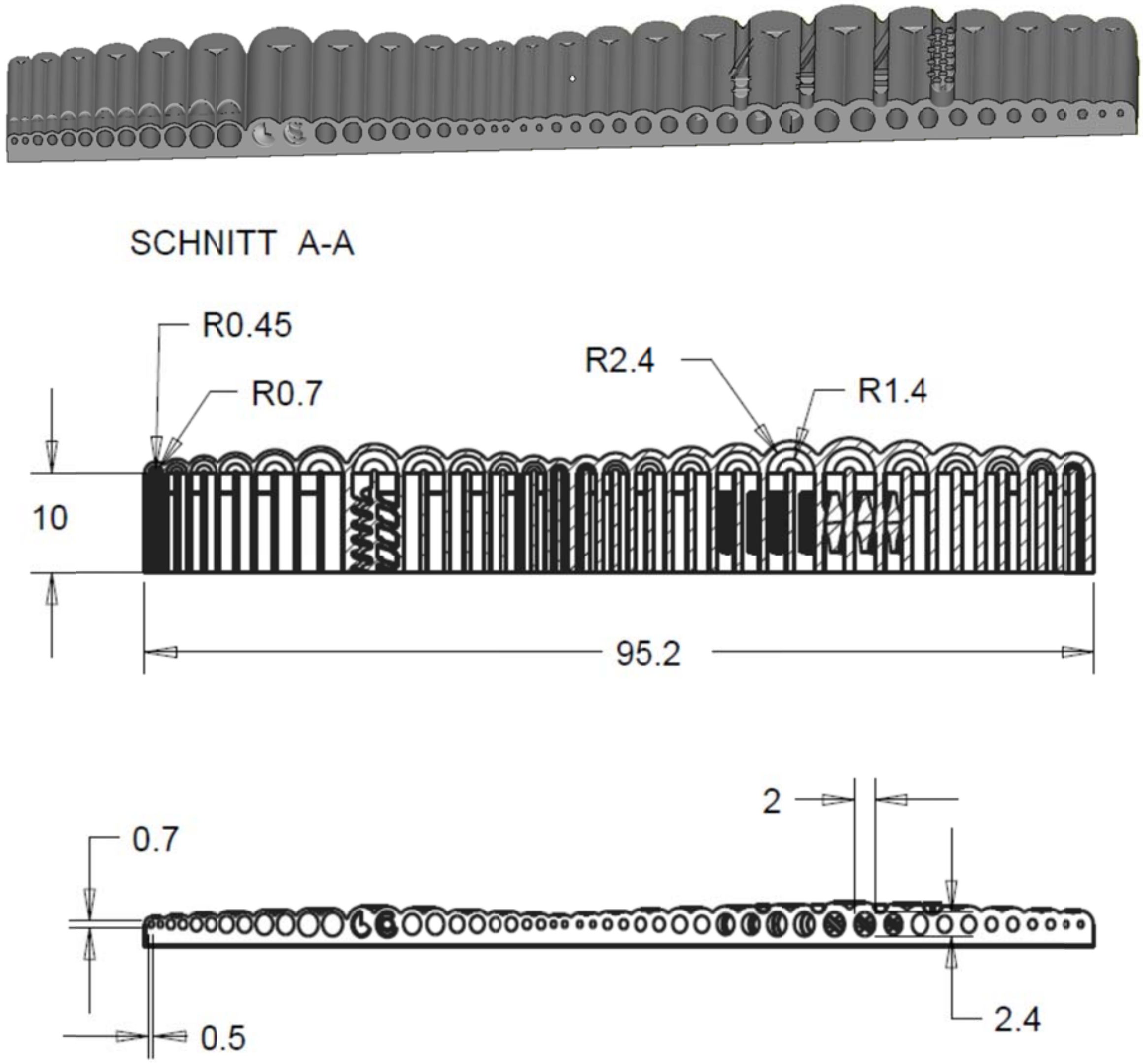


Fig. S1 3D CAD drawing for test print (for the print see Fig. S2) .

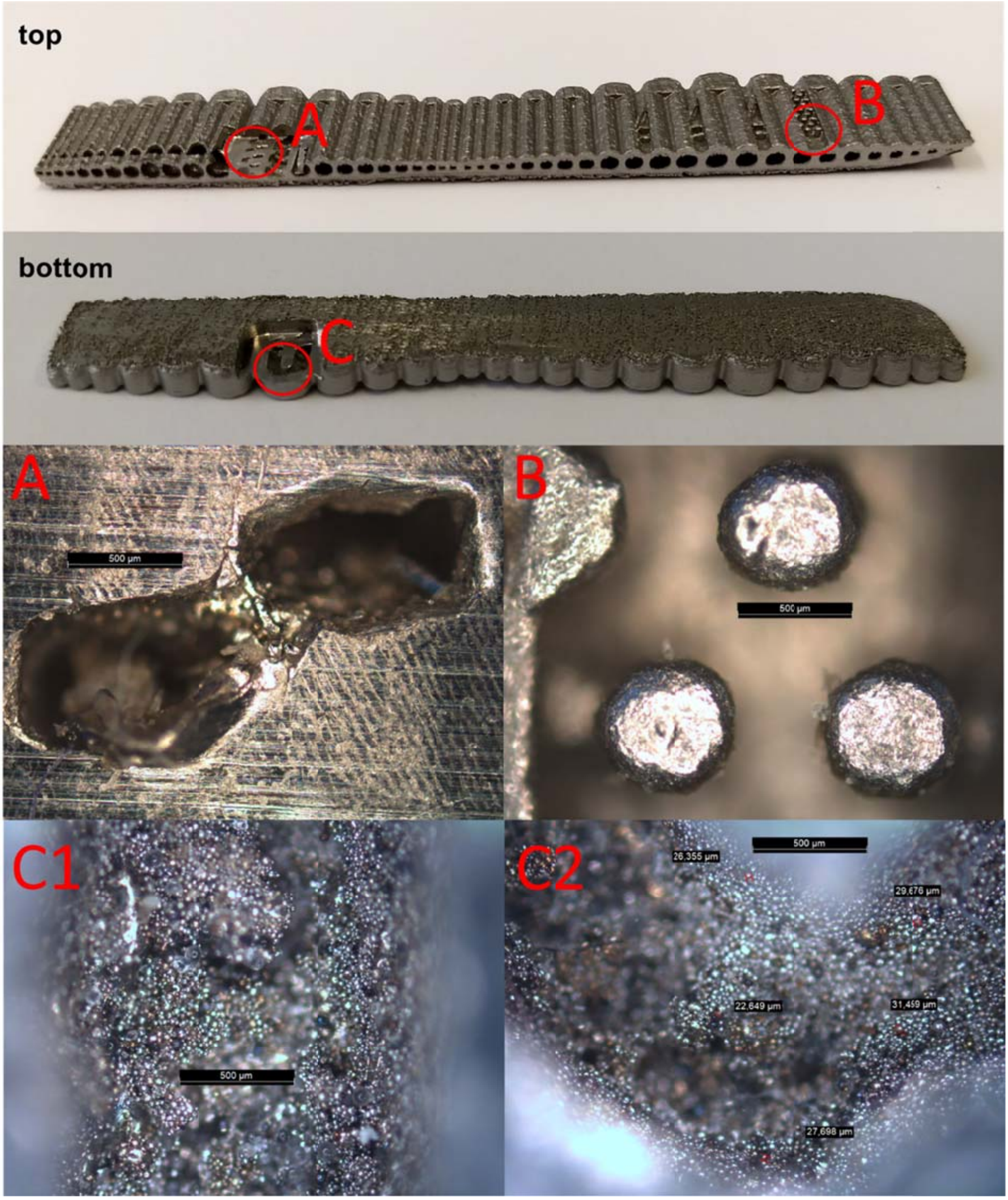


Fig. S2 Microscope pictures of the test print using 316L stainless steel powder. (C1 and C2) Unmolten particles are fused on the top surface of the channel.

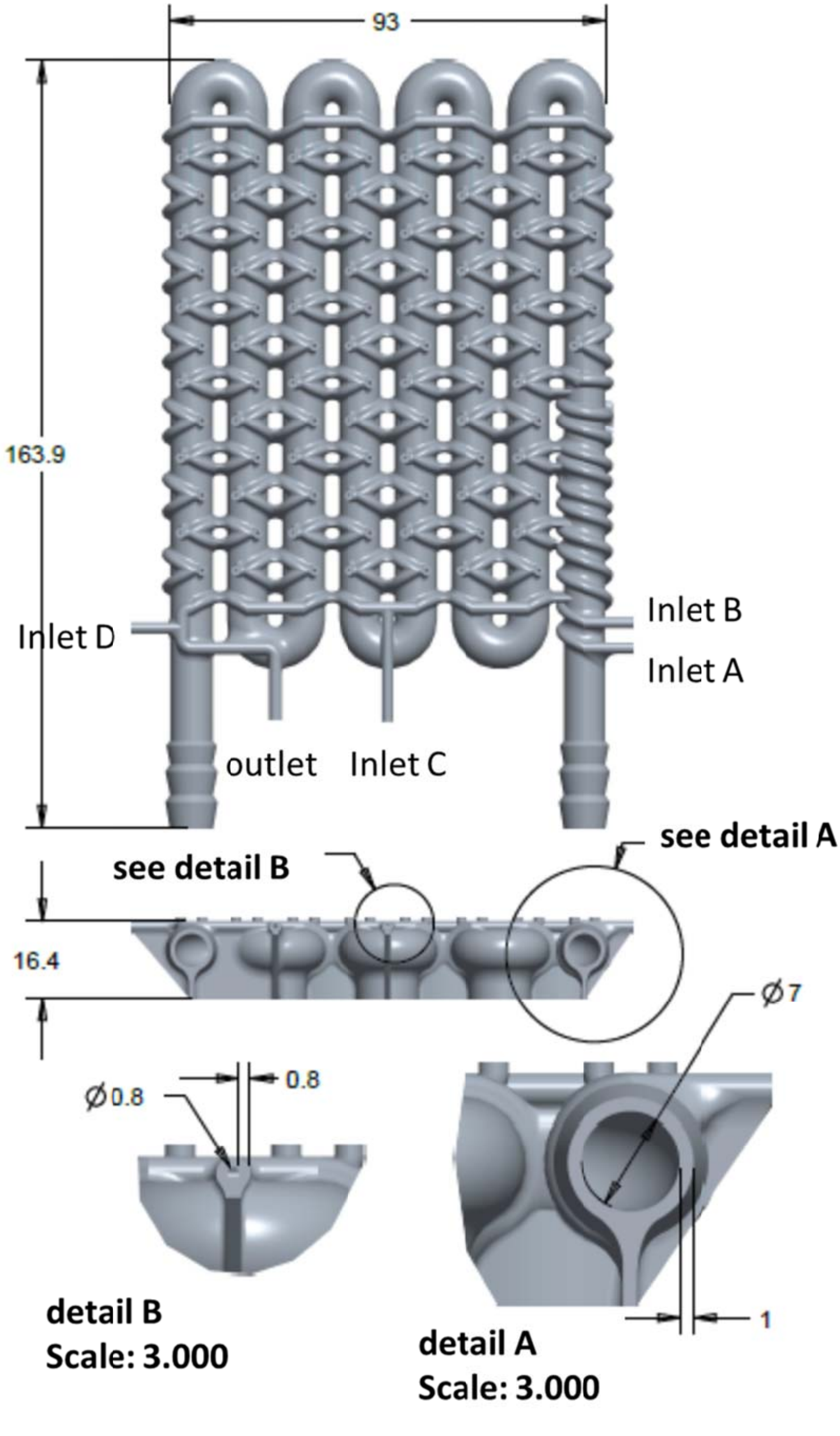


Fig. S3 3D CAD drawing of the continuous flow reactor.

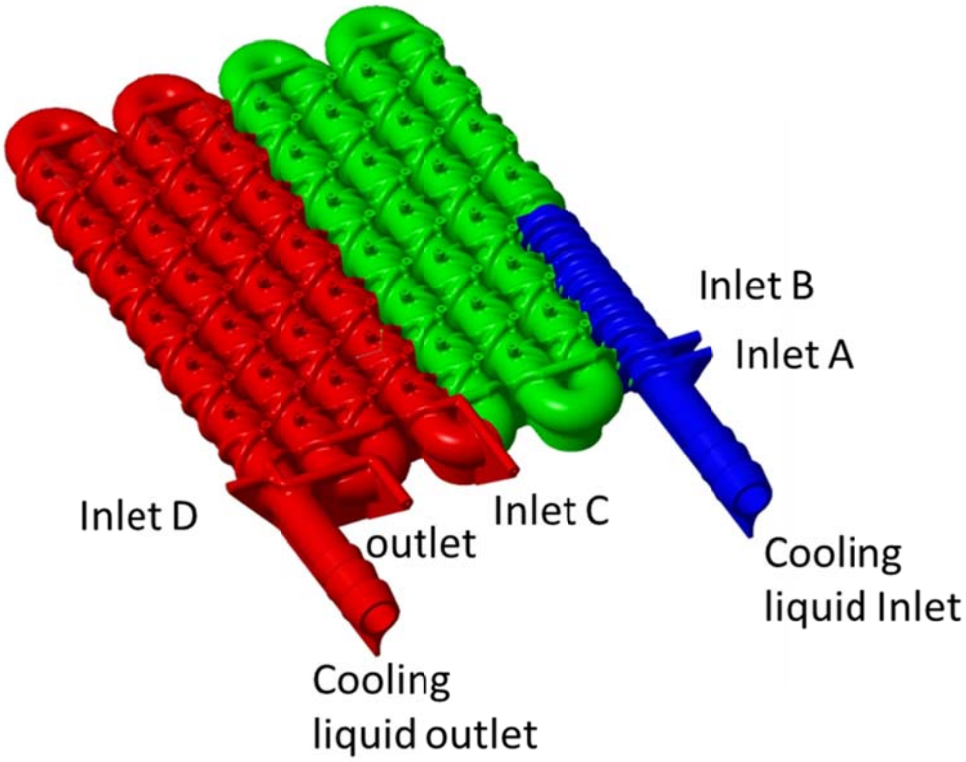


Fig. S4 3D CAD drawing of the continuous flow reactor. Blue: Pre-cooling of feed A and B. Green: reaction of feed A and B. Red: Reaction with feed C.

Fig. S5 EOS DMLS System (for details see: http://www.shapetec.at/kompetenzen/metall-laser-sintern (accessed October 20, 2017)).

Table S1 Particle size distribution characteristics of the 316L stainless steel powder.

Analysis			
Particle	Stainless steel	Particle refraction index	2.757
Dispersion medium	Dry dispersion	Refraction index dispersion medium	1.000
Particle absorption	3.792	Laser Shading	0.89%
Weighted deviation	1.00%	scattering model	Mie
Analysis model	universal	Sensitivity	extented
Results			
Concentration	0.0046%	Width	0.875
Uniformity	0.270	Uits	Volumes
Specific surface	18.69 m^2/Kg	Dv10	28.1 µm
D[3,2]	41.1 µm	Dv50	43.5 µm
D[4,3]	45.7 µm	Dv90	66.2 µm

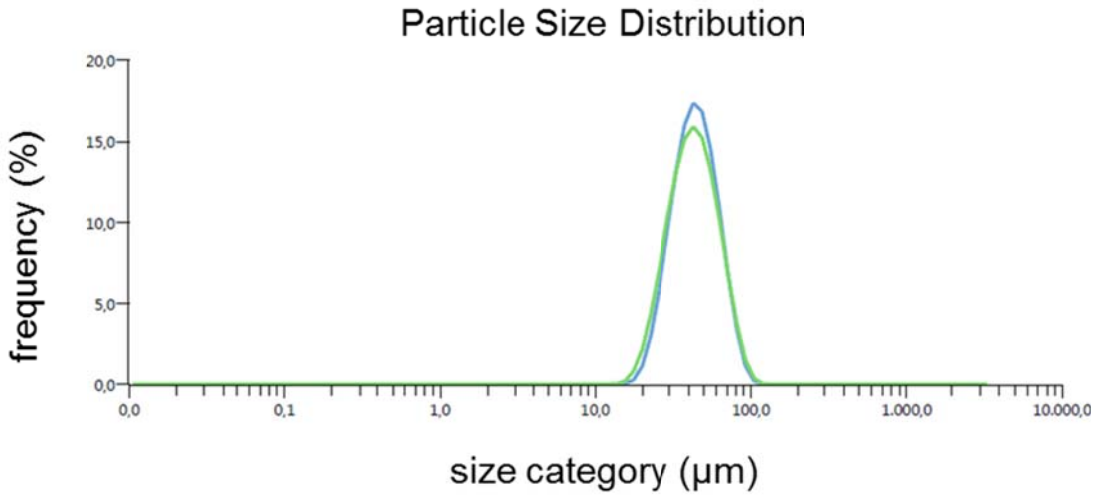


Fig. S6 Particle size distribution characteristics of the 316L stainless steel powder.

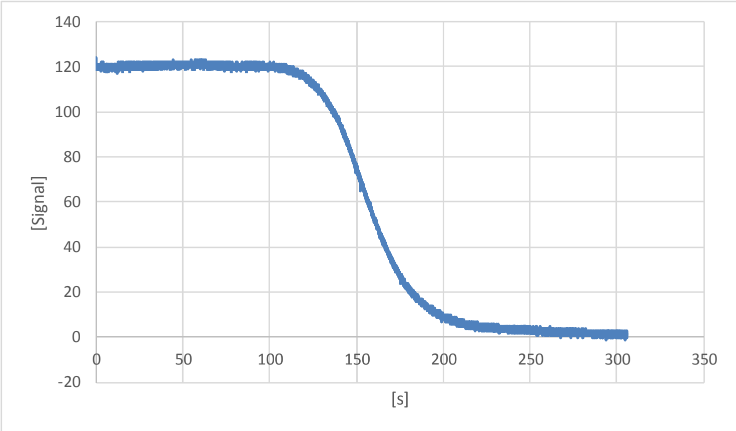


Fig. S7 Step experiment: To determine the residence time distribution (RTD) in the reactor, a step experiment was performed. A 10^{-3} M aqueous solution of Rose Bengal was pumped through the reactor with a flow rate of 1 mL/min. A fast switch from the Rose Bengal solution to water was carried out by means of a three-way valve, while maintaining a flow rate of 1 mL/min. The concentration of Rose Bengal at the outlet was measured in-line with an in-house developed photomete .

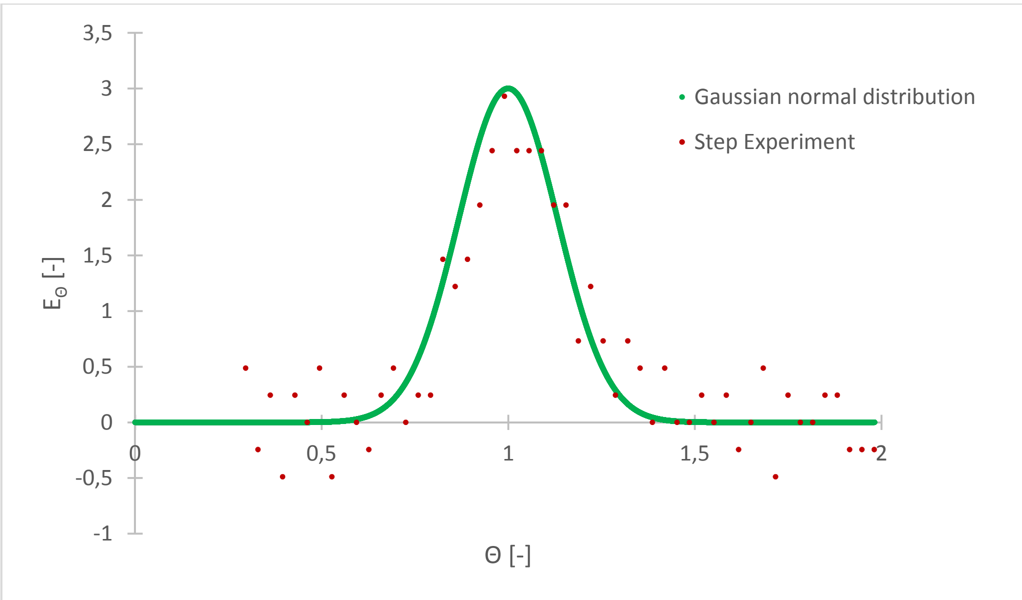
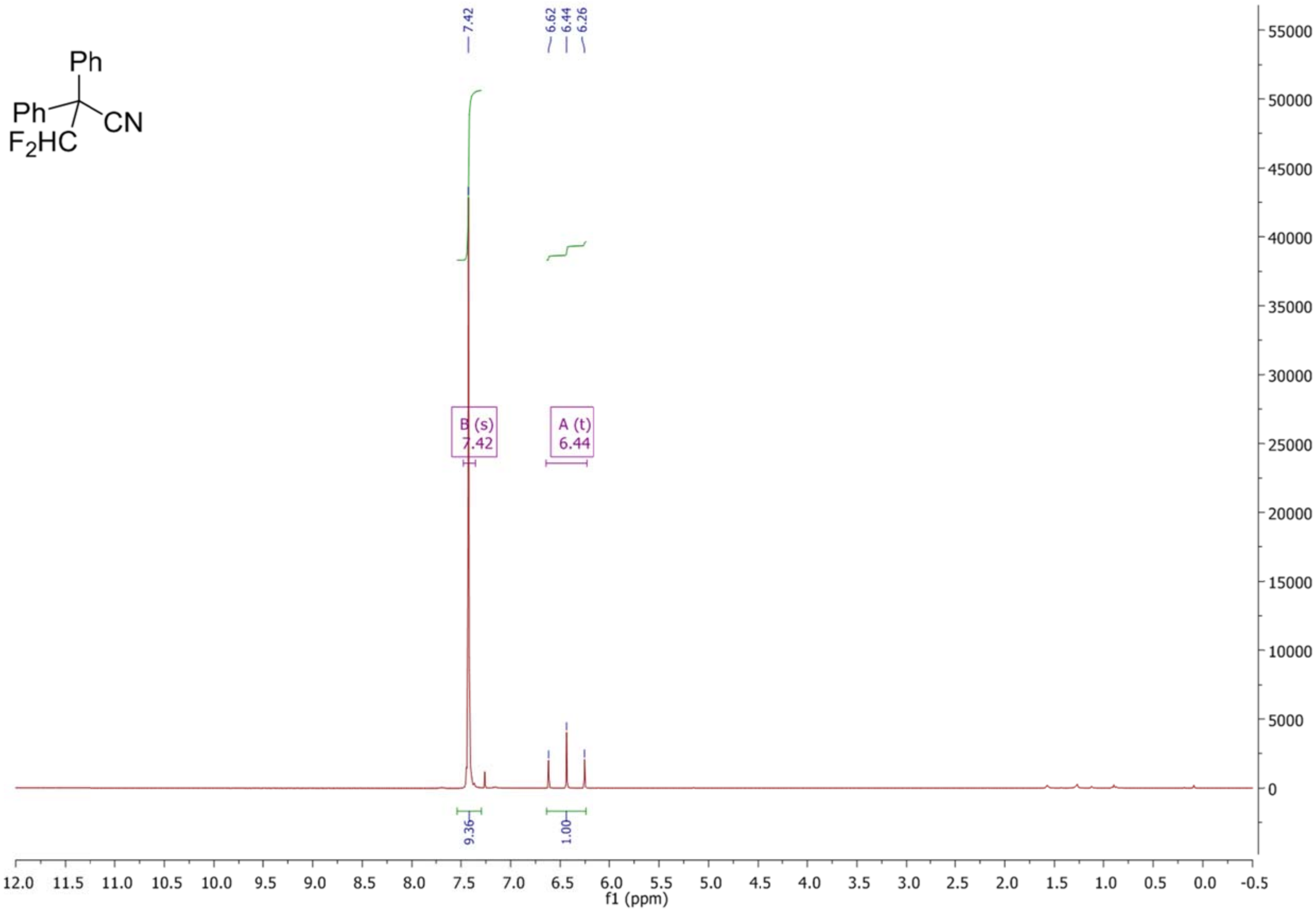
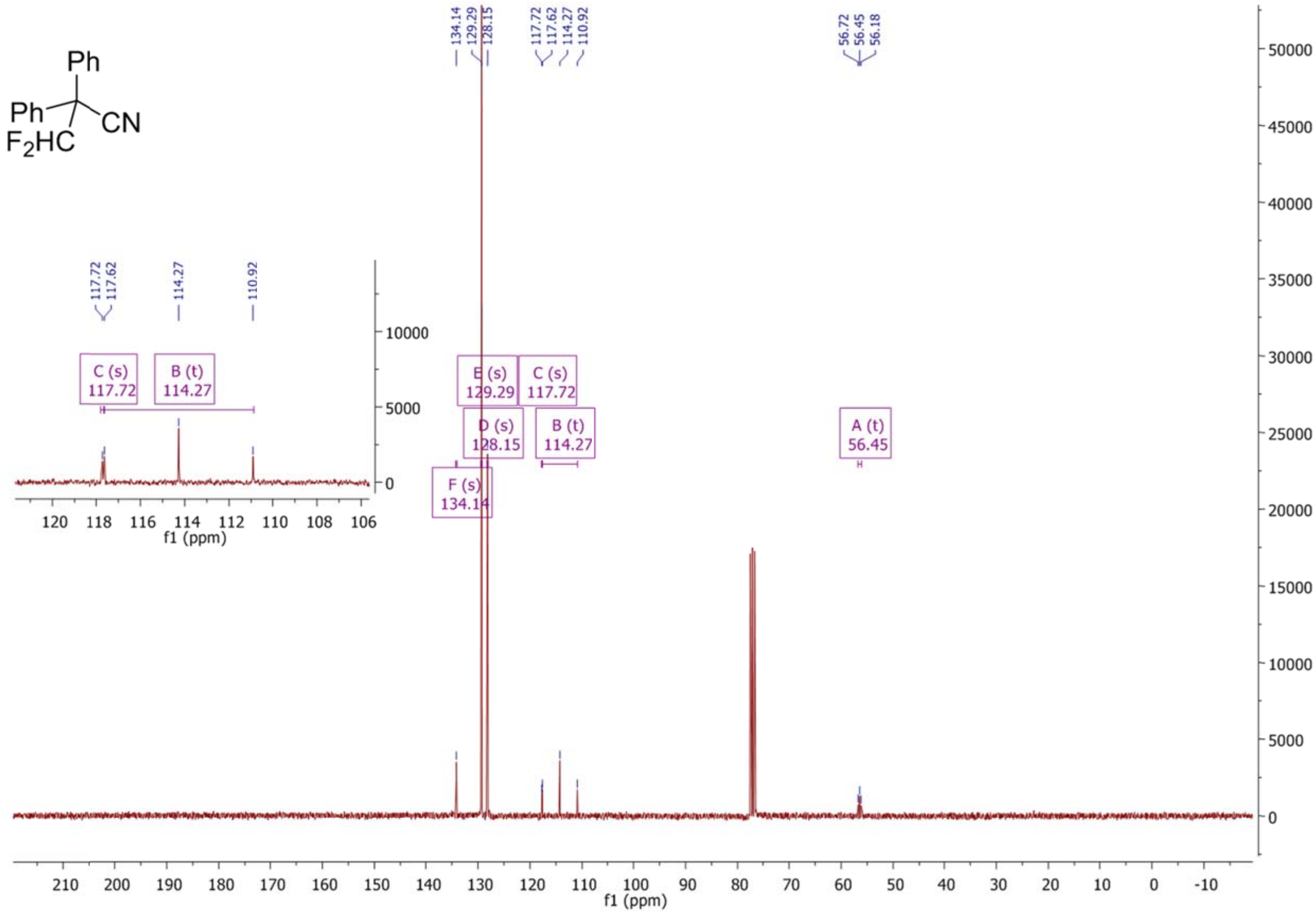


Fig. S8 Determination of the retention time: Dimensionless exit age distribution E_Θ vs. dimensionless time Θ (derived from data points presented in Fig. S7). The evaluation of the experiment was carried out according to the dispersion model by Levenspiel [Levenspiel, O., 1999. Chemical Reaction Engineering. Wiley, New York]. Exit age distribution in dimensionless form of the experiment and a fit with a Gaussian normal distribution can be seen. With the experimental data a Bodenstein number for the open-open vessel condition was found to be 31.44 and mean residence time was calculated to be 154.4 s (reactor plus feed lines) for the step experiment. The Bodenstein number and the narrow exit age distribution suggest moderate axial dispersion throughout the reactor, without channeling or unexpected dead volumes.

^{1}H-NMR of difluoromethyldiphenylactonitrile ($CDCl_3$)



^{13}C-NMR of difluoromethyldiphenylactonitrile ($CDCl_3$)



^{19}F-NMR of difluoromethyldiphenylactonitrile ($CDCl_3$)