

***N*→*O*-Acyl shift in Fmoc-based synthesis of phosphopeptides**

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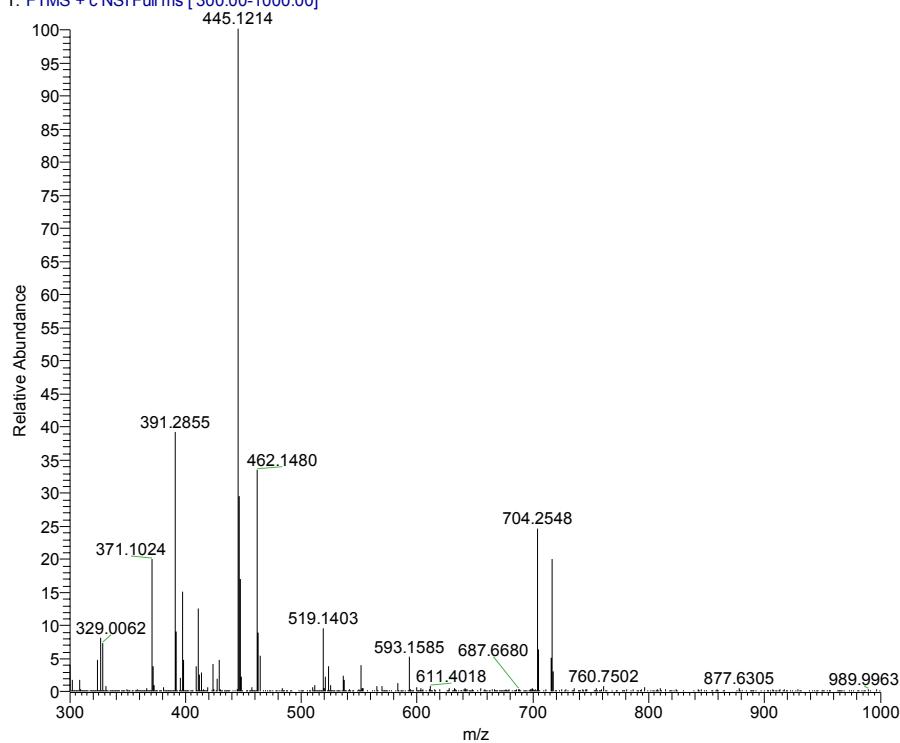
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2. HMQC- and HMBC-spectra of peptide **4a** and **4b**
3. HPLC-traces of peptides **8-11**
4. HPLC-trace of truncation product within synthesis of peptide **12a**

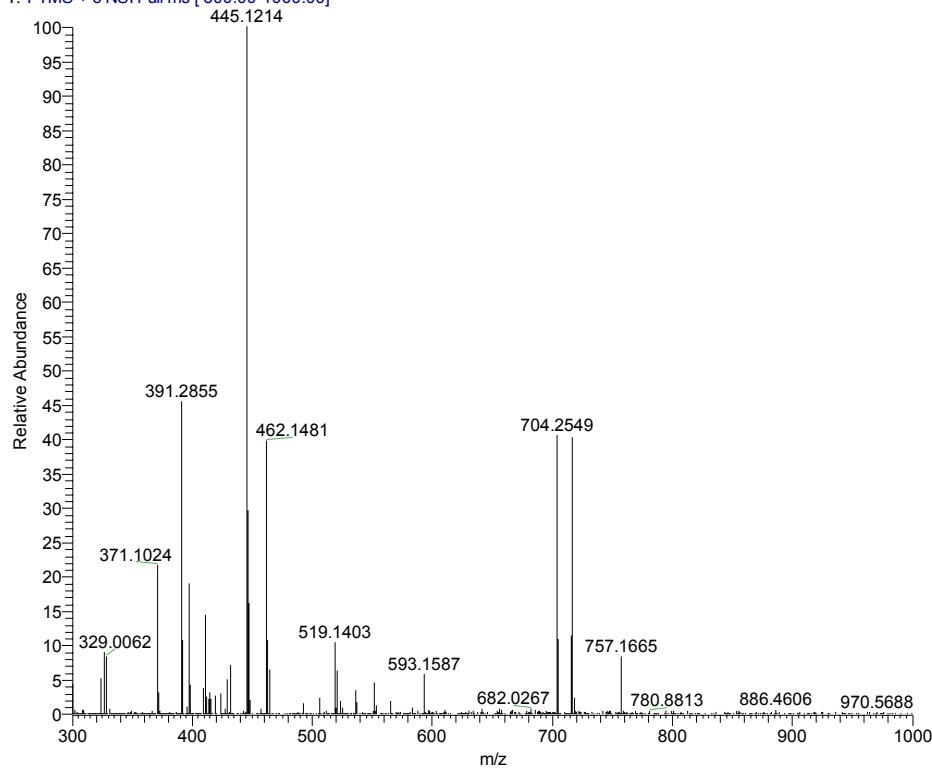
Supplementary Material (ESI) for Organic and Biomolecular Chemistry  
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Hendrik\_Peptid\_5pmol #811-851 RT: 24.68-25.87 AV: 41 NL: 7.86E3  
T: FTMS + c NSI Full ms [ 300.00-1000.00]

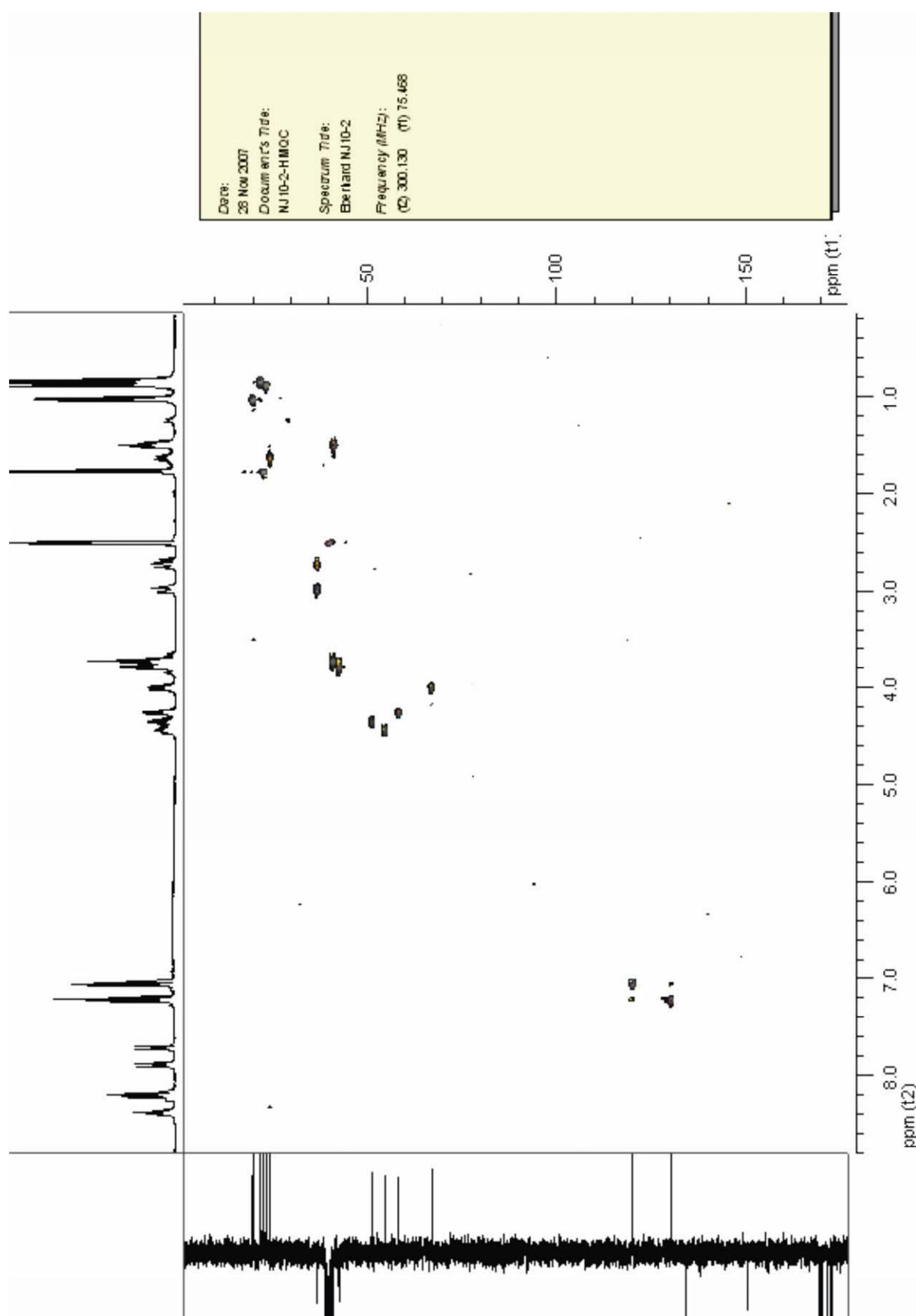


**Fig. S1a** Mass-spectrum of peptide **1b** performed on Finnigan LTQ-FT, ThermoElectron (Bremen), Nano Electrospray, positive ionisation.

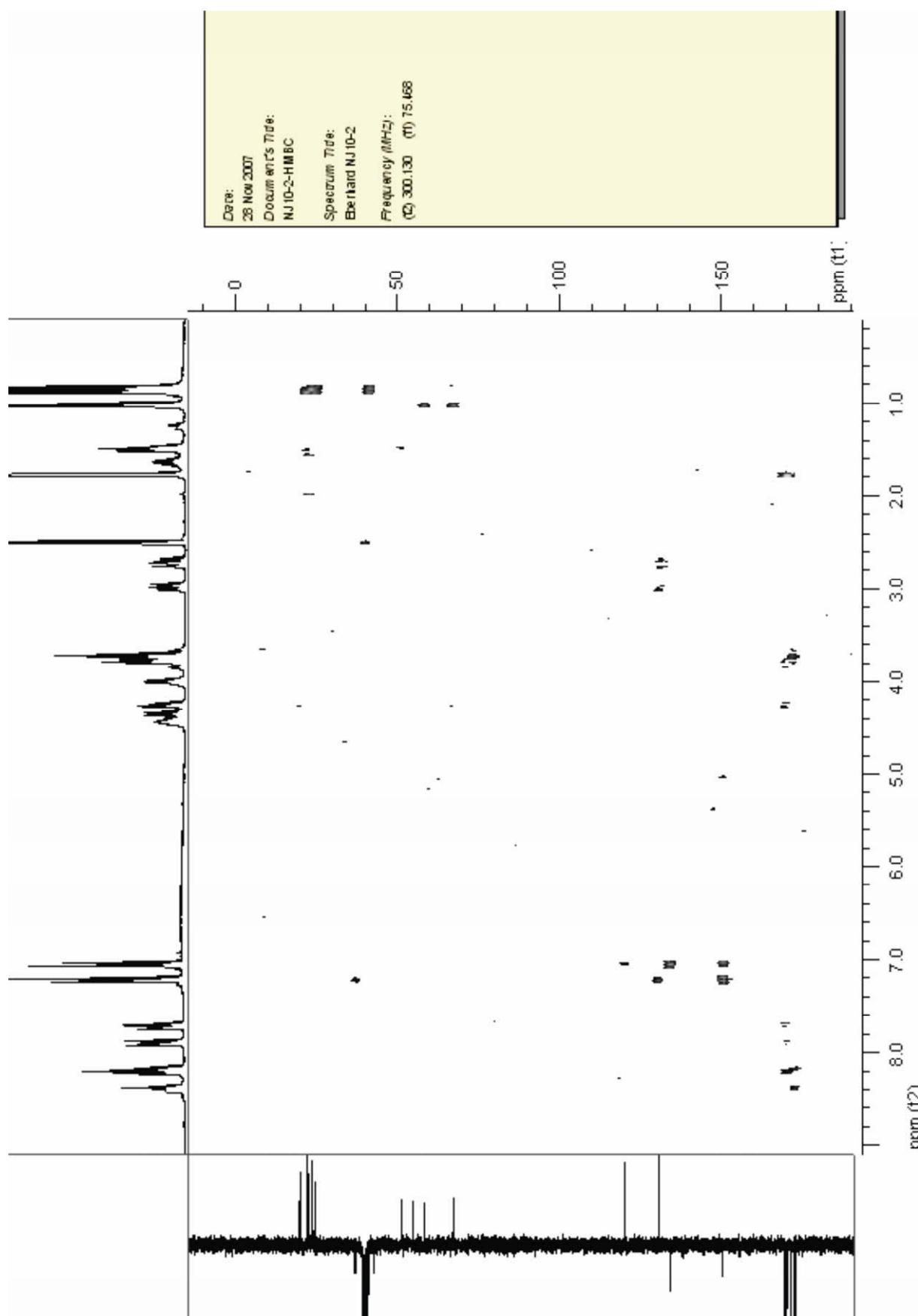
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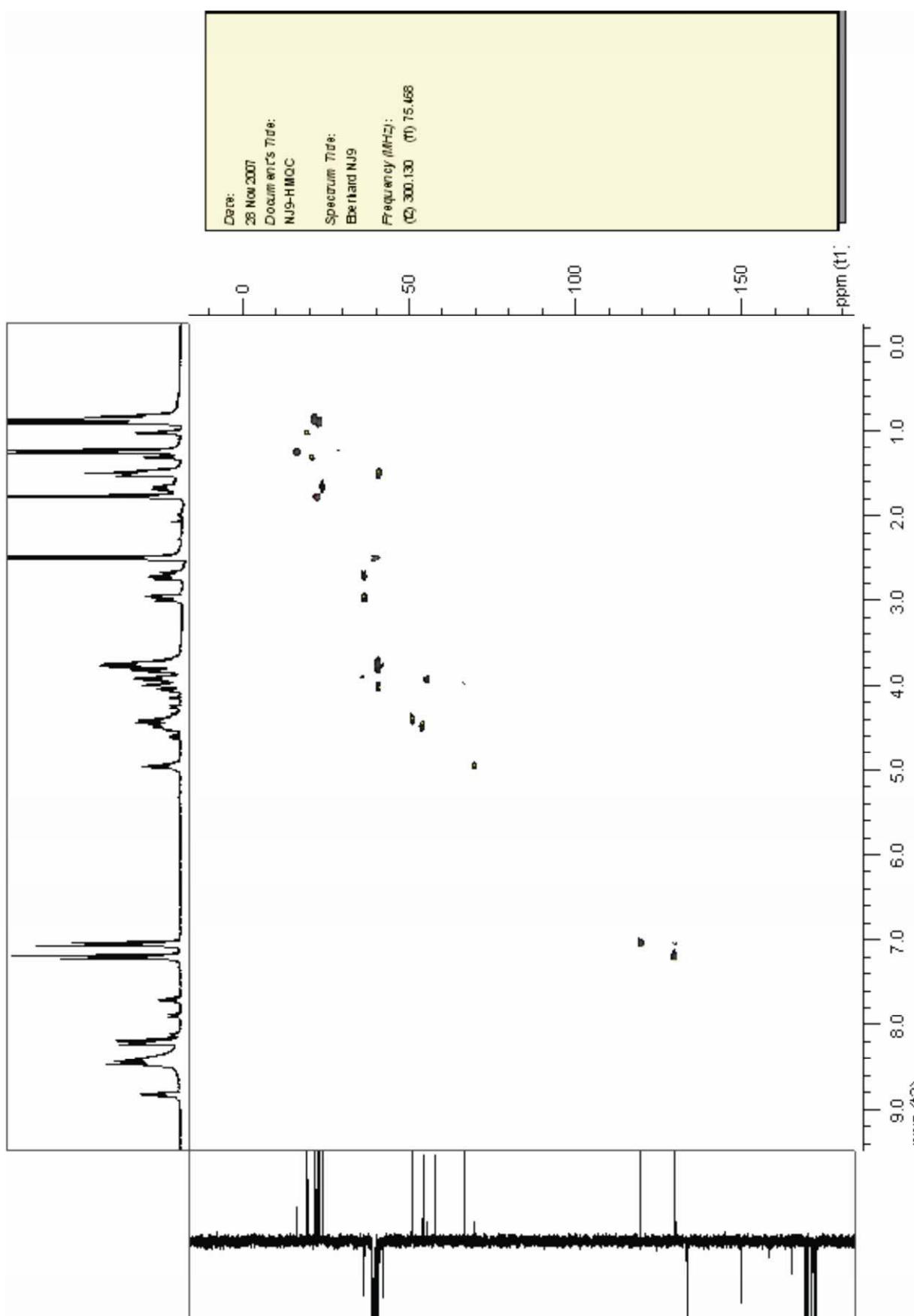
**Fig. S1b** Mass-spectrum of peptide **1a**.



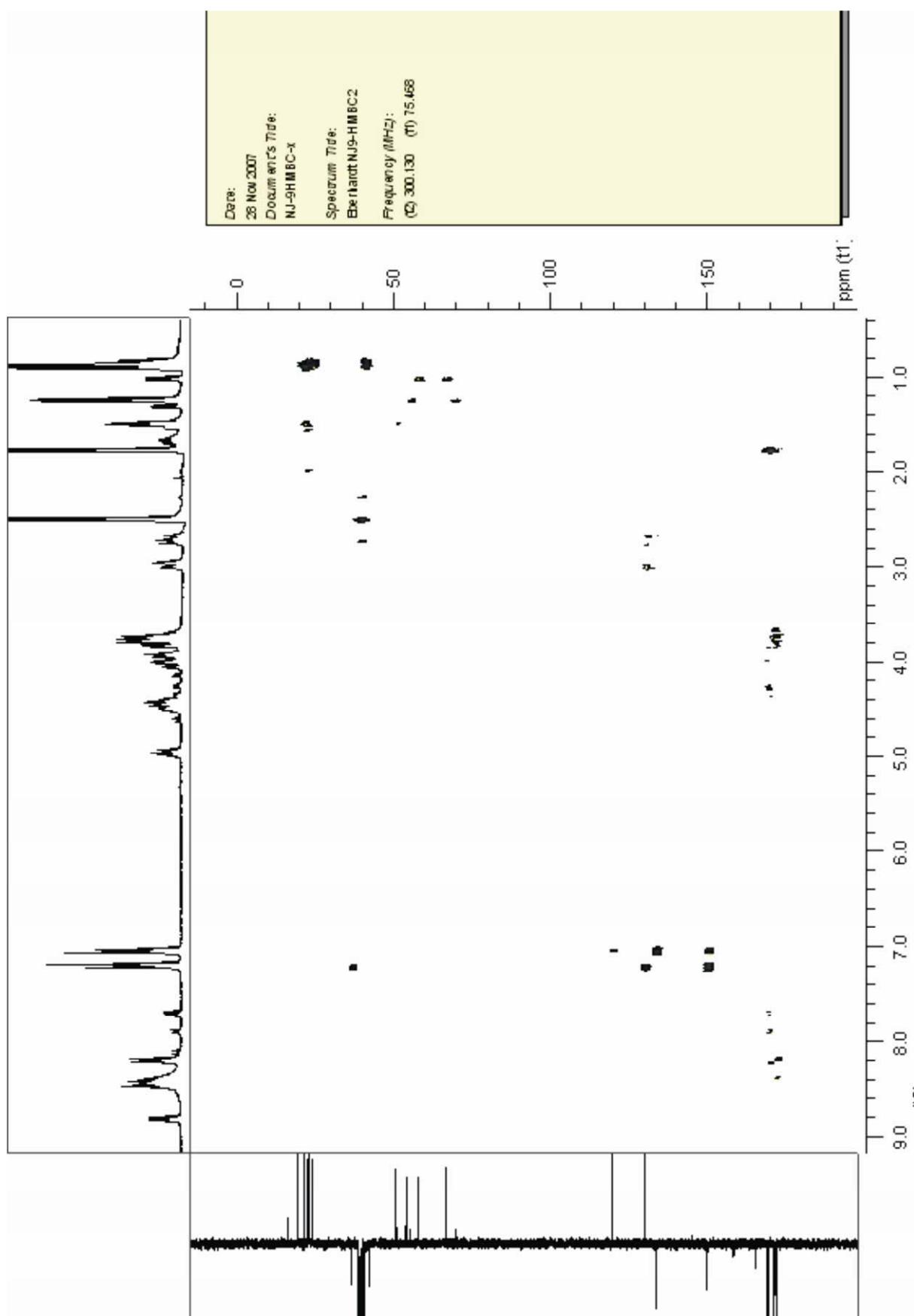
**Fig. S2a** HMQC of peptide **4a**.



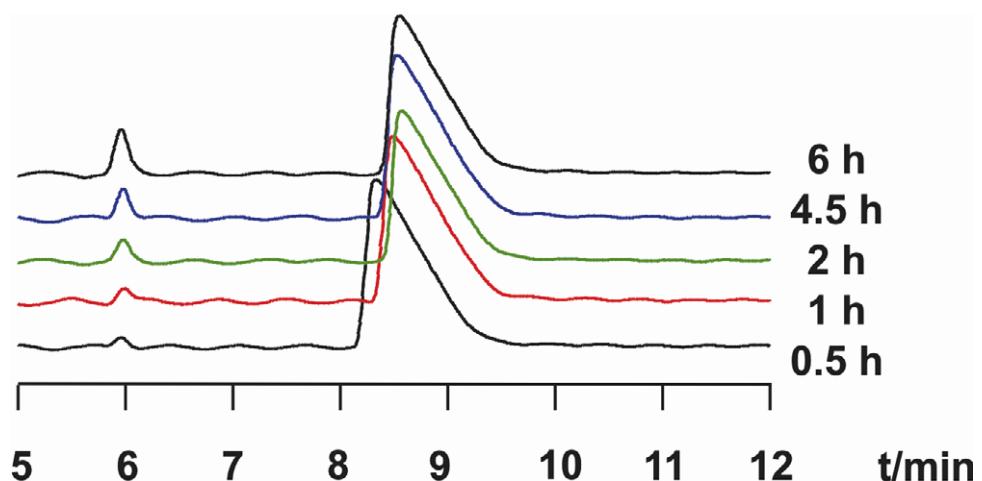
**Fig. S2b** HMBC of peptide 4a.



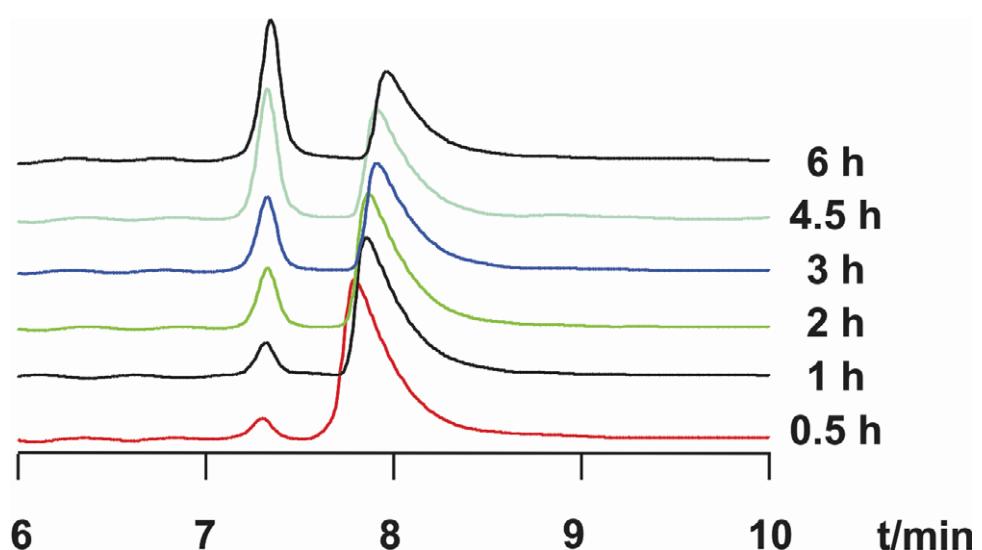
**Fig. S2c** HMQC of peptide **4b**.



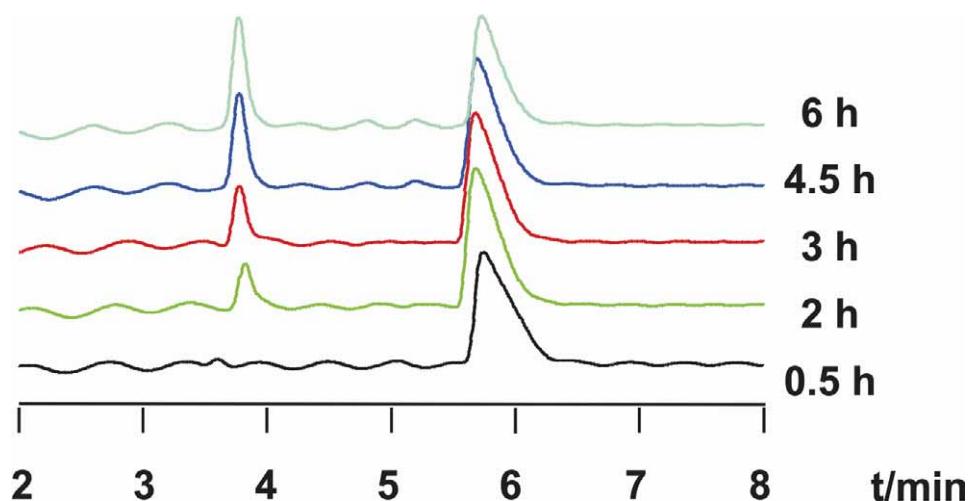
**Fig. S2d** HMBC of depsipeptide **4b**.



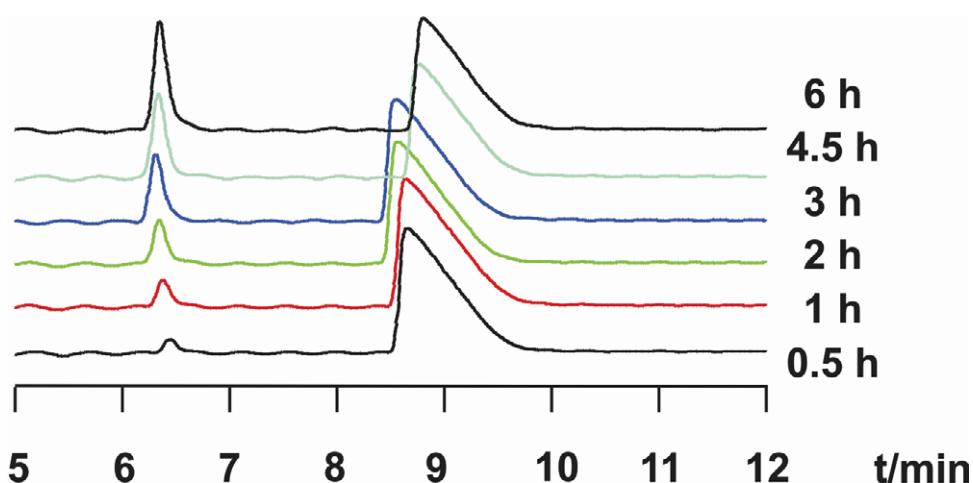
**Fig. S3a** HPLC-traces of depsipeptide formation of peptide **8**.



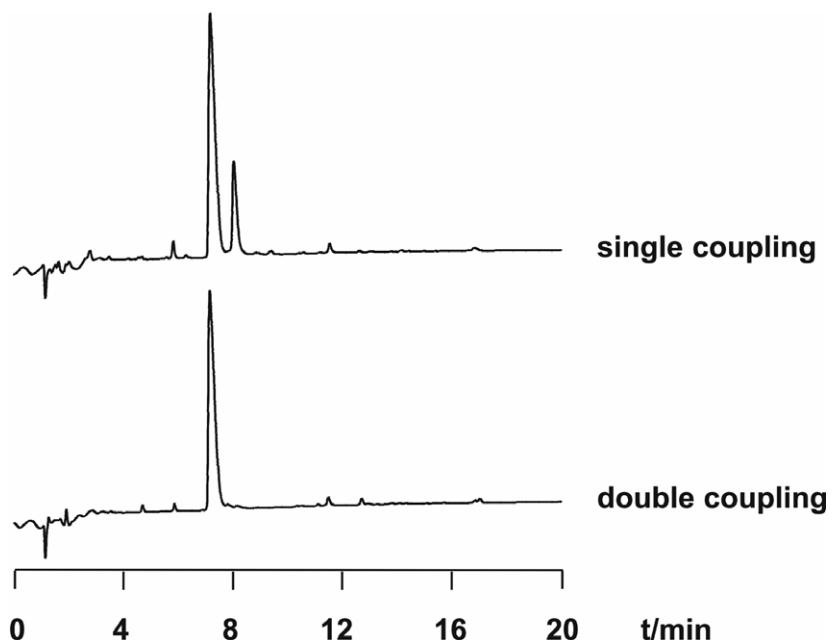
**Fig. S3b** HPLC-traces of depsipeptide formation of peptide **9**.



**Fig. S3c** HPLC-traces of depsipeptide formation of peptide **10**.



**Fig. S3d** HPLC-traces of depsipeptide formation of peptide **11**.



**Fig. S4** HPLC-traces of peptide **12a** synthesised with building block **15**.