



University of Groningen

Synthesis of lactams using enzyme-catalyzed aminolysis

Stavila, E.; Loos, Katja

Published in: **Tetrahedron Letters**

DOI: 10.1016/j.tetlet.2012.10.133

IMPORTANT NOTE: You are advised to consult the publisher's version (publisher's PDF) if you wish to cite from it. Please check the document version below.

Document Version Publisher's PDF, also known as Version of record

Publication date: 2013

Link to publication in University of Groningen/UMCG research database

Citation for published version (APA): Stavila, E., & Loos, K. (2013). Synthesis of lactams using enzyme-catalyzed aminolysis. Tetrahedron Letters, 54(5), 370-372. DOI: 10.1016/j.tetlet.2012.10.133

Copyright Other than for strictly personal use, it is not permitted to download or to forward/distribute the text or part of it without the consent of the author(s) and/or copyright holder(s), unless the work is under an open content license (like Creative Commons).

If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.

Downloaded from the University of Groningen/UMCG research database (Pure): http://www.rug.nl/research/portal. For technical reasons the number of authors shown on this cover page is limited to 10 maximum.

Supplementary Data

Synthesis of lactams using enzyme-catalyzed aminolysis E. Stavila, K. Loos*

Polymer Chemistry Department, Zernike Institute for Advanced Materials University of Groningen, Nijenborgh 4, 9747 AG Groningen, The Netherlands



Figure. S1. ¹H NMR spectrum of butyrolactam



Figure. S2. ¹H NMR spectrum of δ -valerolactam



Figure. S3. ¹H NMR spectrum of *E*-caprolactam



Figure. S4. ¹H NMR spectrum of dimer and trimer lactams



Figure. S5. ¹H NMR spectrum from crude product of reaction between 4-aminobutanoic acid with 5-aminovaleric acid



Figure. S6. ¹H NMR spectrum from crude product of reaction between 4-aminobutanoic acid with 6-aminocaproic acid



Figure. S7. ¹H NMR spectrum from crude product of reaction between 5-aminovaleric acid with 6-aminocaproic acid



Figure. S8. ¹H NMR spectrum from crude product of reaction between 4-aminobutanoic acid with 8-aminooctanoic acid



Figure. S1. ¹H NMR spectrum from crude product of reaction between 5-aminovaleric acid with 8-aminooctanoic acid



Figure. S10. ¹H NMR spectrum from crude product of reaction between 6-aminocaproic acid with 8-aminooctanoic acid



Figure. S11. Mass spectrometry (ESI) chromatogram of dimer and trimer lactams

Transesterification assay: A mixture of deactivated N435 (10 mg) and toluene (20 mL) was stirred at 40 °C, and a solution of 4-nitrophenyl actetate (5 mL, 7.25 mmol L⁻¹) in toluene was added. Immediately methanol (6 μ L) was added to the mixture. After 15 minutes, three samples were taken from the mixture and filtered to remove N435 beads. Of each sample, 0.5 mL of the filtrate was dissolved in 9.5 mL toluene, and the resulting solution was used for the UV-absorption measurement. ε pNP (toluene, 304 nm) = 9537 M⁻¹ cm⁻¹ and ε pNPA (toluene, 304 nm) = 2703 M⁻¹ cm⁻¹.

Synthetic activity assay: A mixture of deactivated N435 (100 mg) and toluene was stirred at 90 °C. ε -Caprolactone (1 mL, 9 mmol) was added and stirred for 5h. After 3d, 2 drops of the reaction mixture were withdrawn, and the conversion of ε -caprolactone was analyzed with ¹H NMR spectroscopy.

N435	Deactivated	Conversion	Transesterification	Synthetic Activity	
	[h]	of <i>ɛ</i> -Caprolactam [%]	Activity	Conversion $[\%]^a$	DPn ^a
		$[nmol PNP min^{-1} mg^{-1}]^a$			
1	4	11	59 ± 13	58	8 ± 2
2	24	11	22 ± 0.2	41	3 ± 1

Table S1. Residual activity of N435 deactivated by heat treatment

^aStandard deviation values were calculated from three replicate experiments.