Supporting information

Optimization of the Microwave Assisted Glycosylamines Synthesis Based on a Statistical Design of Experiments Approach

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1-*Amino*-1-*deoxy*-β-D-N-acetylgalactoside (**Am-I-01**). ¹H-NMR (D₂O, 400 MHz): δ 5.21 (d, J = 3.6 Hz, 0.04H α-H1 (starting material)), 4.60 (d, J = 7.6 Hz, 0.05 H, β-H1 (starting material)), 4.30-3.51 (m, 6 H), 4.06 (d, J = 9.2 Hz, 0.64 β-H1), 2.03 (s, 3 H); ESI MS, calcd. for C₈H₁₆N₂O₅: [M + H]⁺ 221.11, found 221.45 [M + H]⁺.



1-*Amino*-1-*deoxy*-β-D-*lactoside* (**Am-II-01**). ¹H-NMR (D₂O, 400 MHz): δ 5.21 (d, J = 3.6 Hz, 0.05 H, α-H1 (starting material)), 4.65 (d, J = 8.0 Hz, 0.06 H, β-H1 (starting material)), 4.43 (d, J = 7.8 Hz, 1 H, β-H7), 4.10 (d, J = 8.8 Hz, 0.84 H, β-H1), 3.97-3.48 (m, 11 H), 3.19 (m, 0.82 H); ESI MS, calcd. for C₁₂H₂₄NO₁₀: [M + H]⁺ 342.14, found 342.46 [M + H]⁺.



1-*Amino*-1-*deoxy*-β-*D*-glucopyranuronoside (**Am-III**). ¹H-NMR (D₂O, 400 MHz): δ 5.24 (d, J = 3.7 Hz,4 H, α-H1 (starting material)), 4.64 (d, J = 7.9 Hz, β-H1 (starting material), 4.09 (d, J = 8.8 Hz, 0.82 H, β-H1), 3.12-4.34 (m, 4 H); ESI MS, calcd. for C₆H₁₂NO₆: [M + H]⁺ 194.07, found 194.43 [M + H]⁺.



1-*Amino*-1-*deoxy*- β -*L*-*fucose* (**Am-IV**). ¹H-NMR (D₂O, 400 MHz): δ 4.15-4.03 (m, 0.26 H), 3.99 (d, *J* = 8.8 Hz, 0.70 H, β -H1), 3.36-3.94 (m, 3.20 H), 3.32 (t, *J* = 8.8 Hz, 0.58 H) 1.20 (d, *J* = 6.4 Hz, 3 H); ESI MS, calcd. for C₆H₁₄NO₄: [M + H]⁺ 164.09, found 164.38 [M + H]⁺.





Figure S1. Overview plot of yields of GalNAcNH2. Replicates are indicated in blue.



Figure S2. Histogram of yields of GalNAcNH2. Skewness test not triggered.



Figure S3. Plot of GalNAcNH₂ with residuals of yields versus the normal probability of the distribution.



Figure S4. Plot of observed values versus predicted values for yields of GalNAcNH2.



Figure S5. Overview plot of yields of LacNH₂. Replicates are indicated in blue.



Figure S6. Histogram of yields of LacNH2. Skewness test not triggered.



Figure S7. Plot of LacNH₂ with residuals of yields versus the normal probability of the distribution.



Figure S8. Plot of observed values versus predicted values for yields of LacNH2.



Figure S9. Overview plot of yields of GlcANH₂. Replicates are indicated in blue.



Histogram - Yield [%]

Figure S10. Histogram of yields of GlcANH₂. Skewness test not triggered.



Figure S11. Plot of GlcANH₂ with residuals of yields versus the normal probability of the distribution.



Figure S12. Plot of observed values versus predicted values for yields of GlcANH₂.



Figure S13. Overview plot of yields of FucNH₂. Replicates are indicated in blue.



Figure S14. Histogram of yields of FucNH₂. Skewness test triggered. No transformation performed.



Figure S15. Plot of FucNH₂ with residuals of yields versus the normal probability of the distribution.



Figure S16. Plot of observed values versus predicted values for yields of FucNH2.