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Synthesis of derivatization reagent TAHS (N,N,N-trimethylamonioanilyl N-hydroxysuccinimidyl carbamate iodide)

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Works for me

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ABSTRACT

TAHS was synthesized according to the protocol previously described with minor modifications. TAHS is used as derivatization reagent for amino acids. The purpose is to generate enough TAHS for on-tissue derivatisation to enhance signals during MALDI-MSI of amino acids from tissue sections.

THIS PROTOCOL ACCOMPANIES THE FOLLOWING PUBLICATION

<http://onlinelibrary.wiley.com/doi/10.1002/ange.201702669/abstract><http://onlinelibrary.wiley.com/doi/10.1002/pmic.201300041/full><http://pubs.acs.org/doi/abs/10.1021/ac900470w>

MATERIALS

NAME	CATALOG #	VENDOR
N,N'-Disuccinimidyl carbonate	225827	Sigma Aldrich
2,5-Dimethyl-1,4-phenylenediamine	336068	Sigma Aldrich
Iodomethane	18507	Sigma Aldrich
Acetonitrile		
Dichloromethane		

SAFETY WARNINGS

Be careful with organic solvents and chemicals, read safety instructions of each chemical you use

BEFORE STARTING

Work has to be done under fume hood

- 6.0 g (23.4 mmol) DSC was dissolved in 250 mL of dry ACN to which 3.0 g (22.0 mmol) DPD in 250 mL dry ACN was added dropwise over a period of 45 min and stirred (Room temperature)

DSC Molecular Weight 256.17 $C_9H_8N_2O_7$

DPD Molecular Weight 136.19 $(CH_3)_2NC_6H_4NH_2$

1.2 g DSC

0.6 g DPD

50 ml ACN

- 2 The product was concentrated using rotary evaporation and resuspended in 5 mL ACN and filtered through Whatman filter paper No.2 Qualitative.

The product should be checked by mass-spectrometry and NMR for purity

m/z 278.1141 [M +H]⁺, molecular formula, C₁₃H₁₆N₃O₄

- 3 The filtrate was concentrated and redissolved in 10 mL 4:1 ACN:DCM after which 0.4 mL iodomethane (8 equiv.) was added and left to react overnight at room temperature.
- 4 The reaction mixture was filtered to afford approx. 0.3 g of TAHS (6.55 mmol, 29.8% yield over 2 steps).
- 5 The product TAHS should be checked by mass-spectrometry and NMR for purity

m/z 292.1297 [M]⁺, molecular formula, C₁₄H₁₈N₃O₄ (Δ -2.9 ppm).



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