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Data Article

Smart tools and orthogonal click-like reactions onto small unilamellar vesicles: Additional molecular data



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ABSTRACT

We present here the synthetic routes and the experimental data (NMR and MS spectra) for model reactions for copper-free Huisgen 1,4-cycloaddition, Staudinger ligation and for addition of a dithiol on a dibromomaleimide ring. Starting materials were synthesized from the commercially available 4-chlorophenethylamine, previously described 2-(cyclooct-2-yn-1-yloxy)acetic acid, 1-fluorocyclooct-2-ynecarboxylic acid, commercial 2-(diphenylphosphino)terephthalic acid 1-methyl 4-pentafluorophenyl diester and dibromomaleimide. In all cases, the expected compounds were obtained with good yield (50% to quantitative). A novel synthesis of the lipid anchor DOGP₃NH₂ is also described. These data were used as basis for the study reported in the article “Smart Tools and Orthogonal Click-like Reactions onto Small Unilamellar Vesicles” in Chemistry and Physics of Lipids [1].

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Specifications table.

Subject area	Chemistry
More specific subject area	Bioconjugation
Type of data	Experimental synthesis protocols, analysis description, NMR and MS spectra
How data was acquired	¹ H NMR spectra at either 300 MHz, 400 MHz or 500 MHz and ¹³ C NMR spectra at either 75 MHz, 100 MHz or 133 MHz recorded on Bruker spectrometers either 300, 400 or 500 respectively with residual undeuterated solvent as internal reference. High-resolution mass spectra (HRMS) obtained using an Agilent Q-TOF (time of flight) 6520 and low-resolution mass spectra (LRMS) using an Agilent MSD 1200 SL (ESI/APCI). Analytical RP-HPLC-MS performed using a C18 column (30 mm × 1 mm; 1.9 μm) using the following parameters: (1) the eluent system A (0.05% TFA in H ₂ O) and B (0.05% TFA in acetonitrile); (2) the linear gradient <i>t</i> =0 min with 98% A, <i>t</i> =5 min with 5% A, <i>t</i> =6 min with 5% A, <i>t</i> =7 min with 98% A, and <i>t</i> =9 min with 98% A; (3) flow rate of 0.3 mL min ⁻¹ ; (4) column temperature 50 °C; (5) ratio of products determined by integration of spectra recorded at 210 or 254 nm; and (6) ionization mode ESI.
Data format	Analyzed data
Experimental factors	Starting compounds were either purchased or synthesized using already published synthetic protocols
Experimental features	Compounds were synthesized and their structure was identified by NMR and confirmed by mass spectrometry
Data source location	Illkirch, France
Data accessibility	Data are provided in the paper

Value of the data

- The data presented prove the efficiency of bioconjugation reactions and allow reproducibility of the syntheses.
- The overall work provides further tools for surface modification of liposomes.
- The article describes a novel, easier synthesis of the lipid anchor DOGP₃NH₂.

1. Data

All the described ¹H NMR and ¹³C NMR spectra, as well as MS spectra, are available as annexes to this article.

¹H NMR spectra at either 300 MHz, 400 MHz or 500 MHz and ¹³C NMR spectra at either 75 MHz, 100 MHz or 133 MHz were recorded on Bruker spectrometers either 300, 400 or 500 respectively with residual undeuterated solvent as internal reference. All chemical shift values (δ), coupling constants (*J*) and the multiplicity (*s*=singlet, *d*=doublet, *t*=triplet, *m*=multiplet, *br*=broad) are quoted in ppm and in Hz, respectively. High-resolution mass spectra (HRMS) were obtained using an Agilent Q-TOF (time of flight) 6520 and low-resolution mass spectra (LRMS) using an Agilent MSD 1200 SL (ESI/APCI).

2. Experimental design, materials and methods

Reagent grade solvents were used without further purification. Polymer supported triphenylphosphine and anhydrous CH₂Cl₂ were purchased from Sigma-Aldrich. The PyBOP was purchased from Novabiochem, the DIEA was from Alfa Aesar and both were used without further purification. Column chromatography was carried out on silica gel 60 (Merck, 70–230 mesh). Analytical RP-HPLC-MS was performed using a C18 column (30 mm × 1 mm; 1.9 μm) using the following parameters: (1) the eluent system A (0.05% TFA in H₂O) and B (0.05% TFA in acetonitrile); (2) the linear gradient *t*=0 min with 98% A, *t*=5 min with 5% A, *t*=6 min with 5% A, *t*=7 min with 98% A, and *t*=9 min with 98% A; (3) flow rate of 0.3 mL min⁻¹; (4) column temperature 50 °C; (5) ratio of products determined by integration of spectra

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