Supplementary material

Oligonucleotide-Peptide Conjugates: Solid-Phase Synthesis in Acidic Conditions and Use in ELISA Assays

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Figure S1. Mass spectrum (electrospray, positive mode) of MBA-p18 peptide. [M] = 1872.68 (expected M= 1873.08). The experimental value of the mass is the average of the values obtained from M$^{2+}$ (1872.52) and M$^{3+}$ (1872.84).

Figure S2. Mass spectrum (electrospray, positive mode) of MBA-CFFCP1 peptide. [M] = 4334.76 (expected M= 4333.20). The experimental value of the mass is the average of the values obtained from M$^{8+}$ (4334.72), M$^{7+}$ (4334.82), M$^{6+}$ (4334.88) and M$^{5+}$ (4334.65).
**Figure S3.** HPLC chromatogram of OPC-1. The desired conjugate eluted in two peaks (two isomers) at around 10.5 min. Initial T8-SH eluted around 9.5 min. Detection wavelength 260 nm.

**Figure S4.** Mass spectrum (MALDI-TOF, negative mode) of OPC-1. [M] = 4435 (expected M= 4438).
Figure S5. HPLC chromatogram of OPC-2. The desired conjugate eluted at around 14 min. Detection wavelength 260nm.

Figure S6. Mass spectrum (MALDI-TOF, negative mode) of OPC-2. [M] = 8208 (expected M= 8211).
**Figure S7.** HPLC chromatogram of OPC-3. The desired conjugate eluted at around 26 min. Detection wavelength 260nm.

![HPLC Chromatogram](image)

**Figure S8.** Mass spectrum (MALDI-TOF, negative mode) of OPC-3. [M] = 10677 (expected M= 10673).

![Mass Spectrum](image)
**Figure S9.** Mass spectrum (electrospray, positive mode) of CFFCP1 linear peptide. [M] = 4141.20 (expected M= 4141.57). The experimental value of the mass is the average of the values obtained from M$^{5+}$ (4141.15), M$^{6+}$ (4141.32), M$^{7+}$ (4141.20) and M$^{8+}$ (4141.28).

**Figure S10.** HPLC chromatogram of OPC-4. The desired conjugate eluted at around 12 min. A) deprotection protocol with TFA and then NH$_3$ and B) deprotection protocol with NH$_3$ and TFA. Detection wavelength 260 nm.

A) ![HPLC chromatogram](image)

B) ![HPLC chromatogram](image)
Figure S11. Mass spectrum (MALDI-TOF, negative mode) of OPC-4. \([M] = 6688\) (expected \(M = 6687\)).

Figure S12. Mass spectrum (electrospray, positive mode) of CFFCP1 Ser peptide. \([M] = 3967.05\) (expected \(M = 3966.28\)). The experimental value of the mass is the average of the values obtained from \(M^4^+\) (3967.32), \(M^5^+\) (3967.0), \(M^6^+\) (3966.9) and \(M^7^+\) (3966.97).
Figure S13. Mass spectrum (MALDI-TOF, negative mode) of OPC-5. [M] = 7820 (expected M= 7814).

Figure S14. HPLC chromatogram of OPC-5. The desired conjugate eluted at around 7.9 min (main peak). Detection wavelength 260nm.
Figure S15. HPLC chromatogram of OPC-6. The desired conjugate eluted at around 11.8 min (main peak). Detection wavelength 260nm.

Figure S16. Mass spectrum (MALDI-TOF, negative mode) of OPC-7 (T12 cyclic). [M] = 7765 (expected M= 7759).
**Figure S17.** Mass spectrum (MALDI-TOF, negative mode) of OPC-8. $[M] = 7686$ (expected M= 7684).

**Figure S18.** HPLC chromatogram of OPC-9. The desired conjugate eluted at around 12 min (main peak). Detection wavelength 260nm.
**Figure S19.** Mass spectrum (MALDI-TOF, negative mode) of OPC-9. $[M] = 7914$ (expected $M= 7918$).

![Mass Spectrum](image1)

**Figure S20.** Analysis of the stability of oligonucleotide-peptide conjugates to TFA. The purified conjugate OPC-9 was treated with TFA / water (95:5 v/v) for 4 hr. The OPC was isolated by precipitation with cold diethyl ether and centrifugation. A) HPLC chromatogram of conjugate OPC-9 before the TFA treatment and B) HPLC chromatogram of conjugate OPC-9 after the TFA treatment. Detection wavelength 260 nm.

![HPLC Chromatograms](image2)