Supporting Information

for

# "Facile One-pot Synthesis of Aptamer-Based Organic-Silica Hybrid Monolithic Capillary Column by "Thiol-ene" Click Chemistry for Detection of Enantiomers of Chemotherapeutic Anthracyclines"

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**Table S1**. Linearities, LODs, and LOQs of doxorubicin and epirubicin spiked in serum and urine from healthy controls analyzed byaptamer-based organic-silica hybrid monolithic capillary column.

| Matrix | Analytes | Regression Line | | R | Linear Range (μg/mL) | LODs | | LOQs | |
|---|---|---|---|---|---|---|---|---|---|
| | | Slope | Intercept | | | In solution(μg/mL) | On column(pg) | In solution(μg/mL) | On column (pg) |
| Serum | Doxorubicin | 1070.1 | 16856.9 | 0.9972 | 5 - 1000 | 0.3 | 10 | 0.8 | 34 |
| | Epirubicin | 1076.7 | 36891.5 | 0.9971 | | 0.3 | 10 | 0.8 | 34 |
| Urine | Doxorubicin | 1057.8 | 28666.0 | 0.9992 | 5 - 1000 | 0.3 | 10 | 0.8 | 34 |
| | Epirubicin | 987.1 | 29974.8 | 0.9988 | | 0.3 | 10 | 0.8 | 34 |

**Table S2.** The recoveries for the determination of doxorubicinand epirubicinspikedin serum and urinefrom healthy controlsat three different concentrations.

| | Recoveries (%, n = 3) | | | | | |
|---|---|---|---|---|---|---|
| | Doxorubicin | | | Epirubicin | | |
| | Low (25 μg/mL) | Medium (100 μg/mL) | High (500 μg/mL) | Low (25 μg/mL) | Medium (100 μg/mL) | High (500 μg/mL) |
| Serum | 94.1 | 90.1 | 91.3 | 93.7 | 93.7 | 90.6 |
| Urine | 94.1 | 97.8 | 95.3 | 93.5 | 90.7 | 96.7 |

**Table S3.** Precisions (intra- and inter-day) for the determination of doxorubicinand epirubicin.

| | Intra-day (RSD %, n = 3) | | | Inter-day (RSD %, n = 3) | | |
|---|---|---|---|---|---|---|
| | Low (25 μg/mL) | Medium (100 μg/mL) | High (500 μg/mL) | Low (25 μg/mL) | Medium (100 μg/mL) | High (500 μg/mL) |
| Doxorubicin | 0.6 | 2.5 | 1.1 | 0.3 | 1.0 | 1.7 |
| Epirubicin | 0.3 | 3.7 | 1.7 | 2.6 | 2.5 | 2.5 |

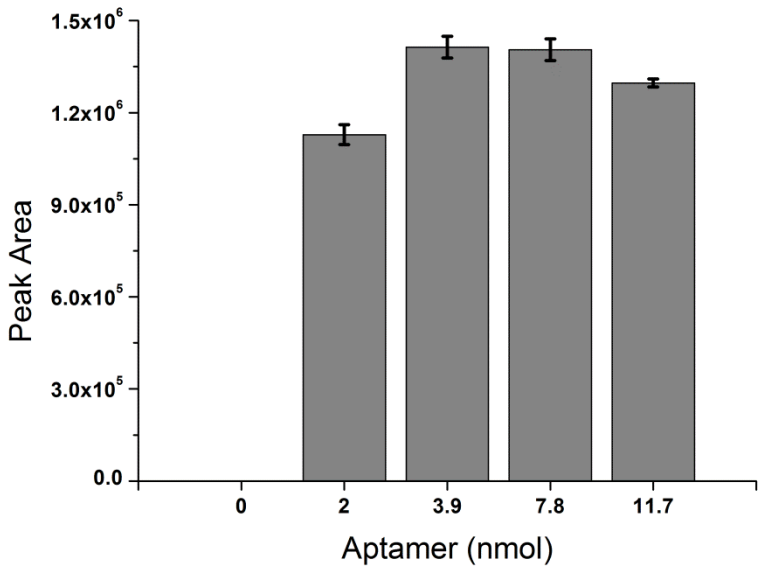


**Figure S1.**Optimization of the amounts of Aptamer-41 in the preparation of aptamer modified monolithic column.Monolithic capillary column: 10cm-long, 75 μmi.d. × 360 μmo.d.; flow velocity, 0.5 mL/min; inject volume, 20μL; doxorubicin, 1000μg/mL; splitting ratio, 1/500; detectionwavelength, 254 nm.

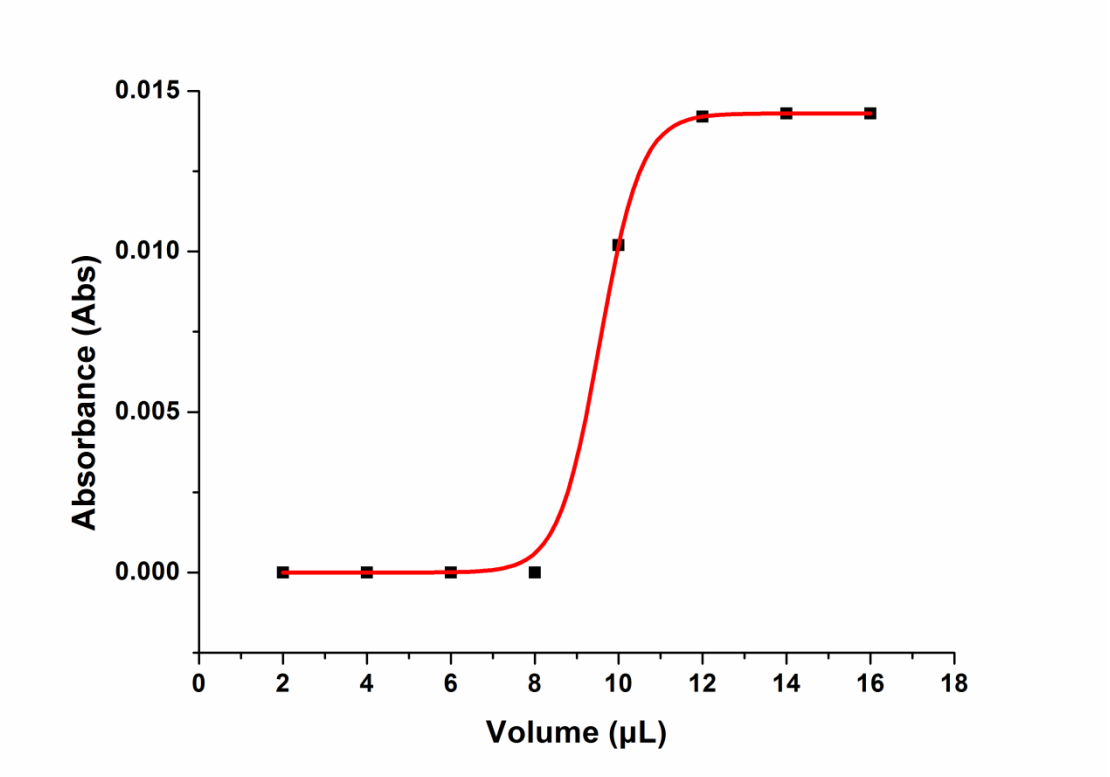


**Figure S2.** Breakthrough curve of doxorubicin in the aptamer-based organic-silica hybrid monolithic capillary column. Monolithic capillary column: 10 cm-long, 75 μm i.d. × 360 μm o.d.; flow rate, 1 μL/min; sample concentration, 10 pmol/μL; detection wavelength, 254 nm.

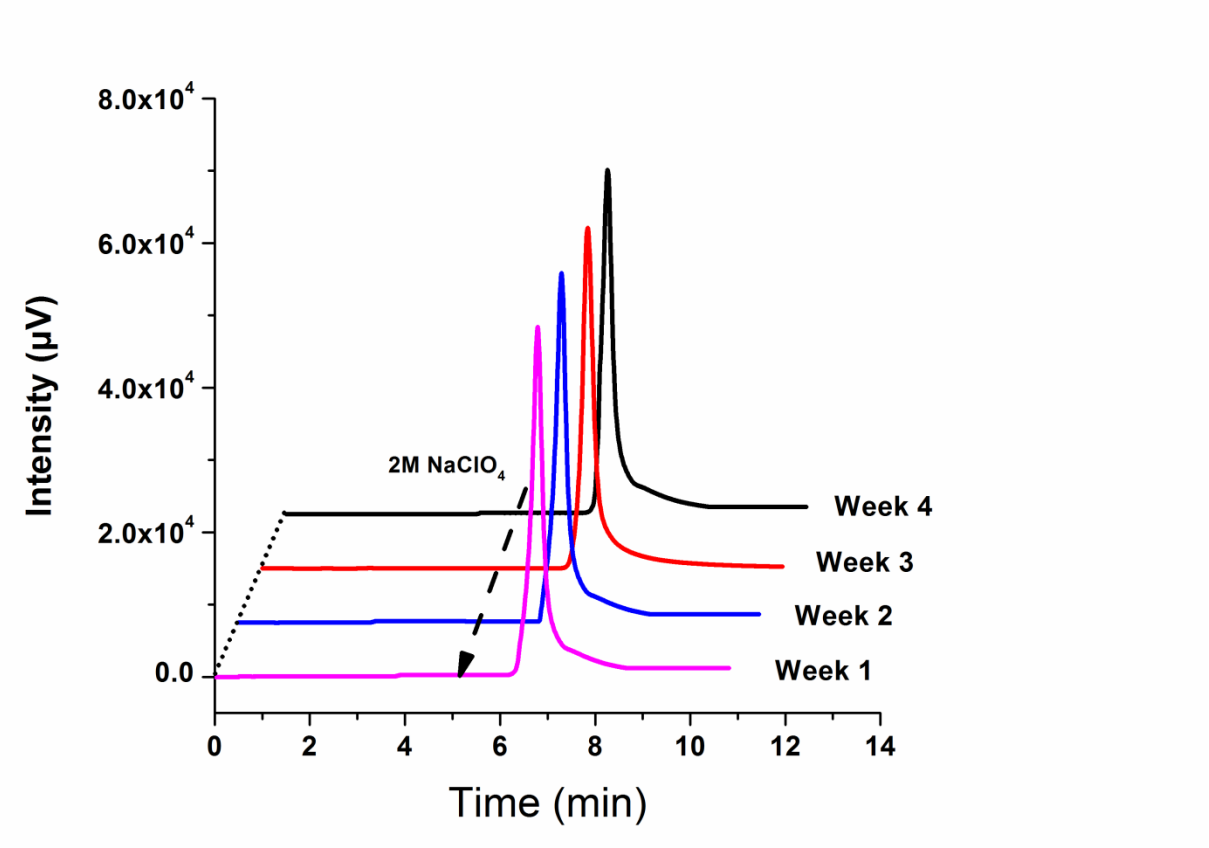


**Figure S3.** Evaluation of the stability of aptamer-based organic-silica hybrid monolithic capillary column by analysis the doxorubicin in consecutive four weeks. Monolithic capillary column: 10 cm-long, 75 μm i.d. × 360 μm o.d.; flow rate, 0.5 mL/min; inject volume, 20 μL; sample concentration, 500 μg/mL; splitting ratio, 1/500; detection wavelength, 254 nm.