

Supplementary Data

Simple and rapid analysis of muscarine in human urine using dispersive micro solid phase extraction and ultra high performance liquid chromatography-high resolution mass spectrometry

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Chromatographic conditions for analysis of muscarine in three different columns.

The aqueous phase (A) consisted of a mixture of 0.1% of formic acid and 4 mM ammonium formate in water, and the organic phase (B) was acetonitrile with 0.1% formic acid.

HILIC column: The gradient started at 98% B was reduced to 80% B in the next 2 minutes and then linearly ramped to 40% B in the following 5 minutes. This was followed by re-equilibration at 98% B for 3 minutes prior to the next injection. The flow rate was set to 300 μ L/min. (Retention time: 4.56 min for muscarine)

C₁₈ column: The gradient started at 5% B for 2 minutes, was raised to 30% B in the next 3 minutes and then linearly ramped to 100% B in the following 1 minutes. This was followed by re-equilibration at 5% B for 3 minutes prior to the next injection. The flow rate was set to 200 μ L/min. (Retention time: 1.21 min for muscarine)

T₃ column: The gradient started at 2% B for 2 minutes, was raised to 50% B in the next 3 minutes and then linearly ramped to 100% B in the following 1 minutes. This was followed by re-equilibration at 2% B for 3 minutes prior to the next injection. The flow rate was set to 200 μ L/min. (Retention time: 2.11 min for muscarine)

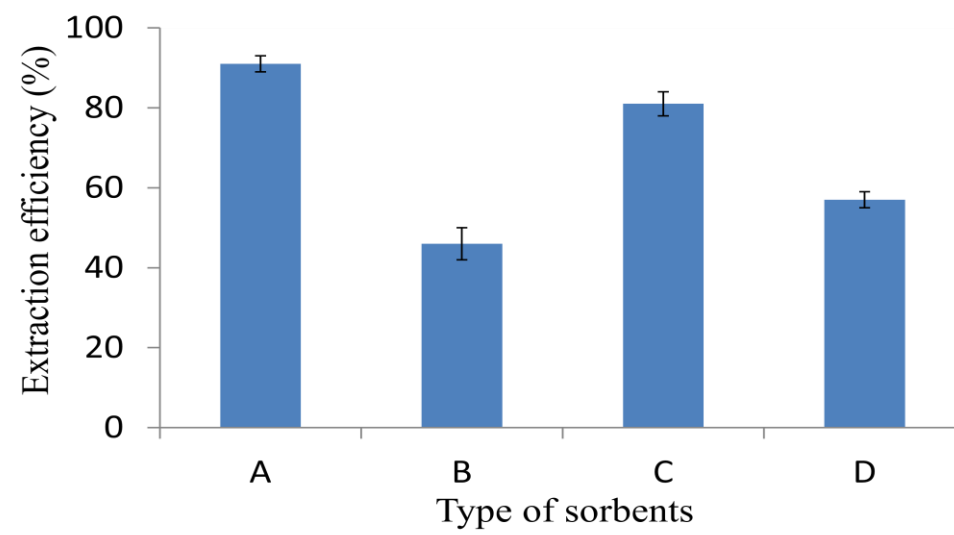


Figure S1 The effect of the different sorbents on the extraction efficiency ($n = 3$). (A) Cleanert® COOH, (B) Cleanert® SCX, (C) Cleanert® PWCX, (D) Cleanert® PCX.

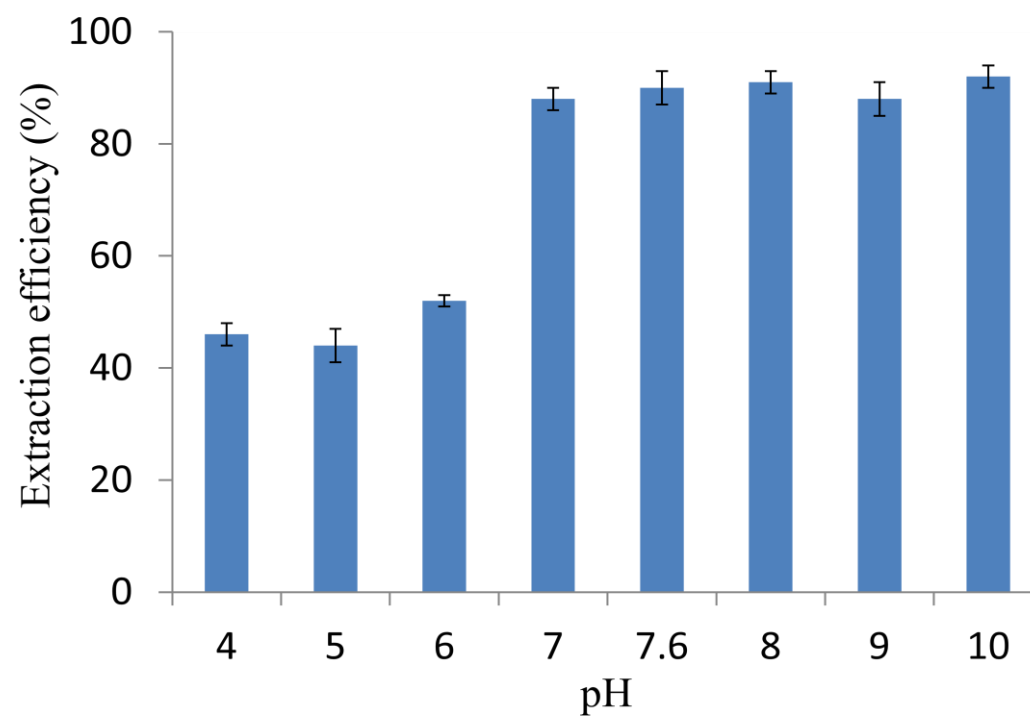
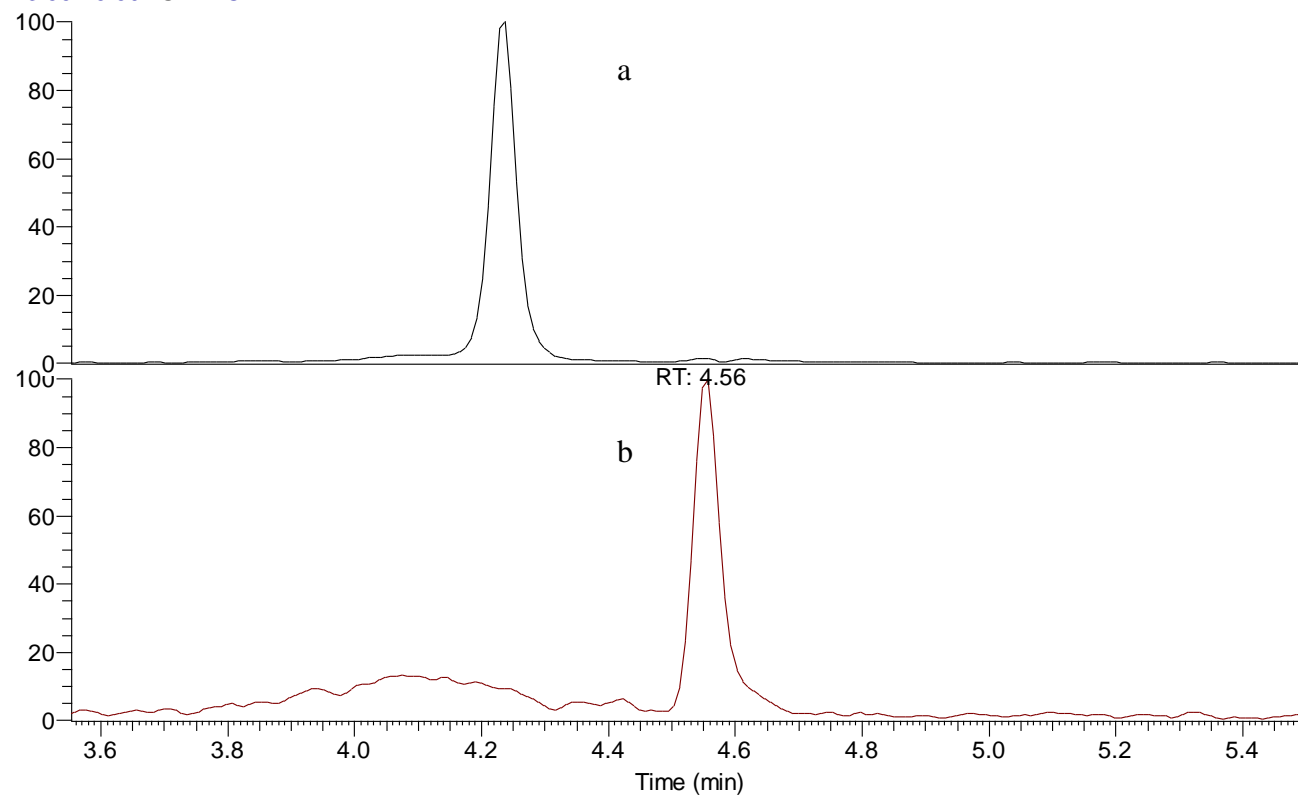


Figure S2 Effect of pH on the extraction efficiency ($n = 3$).

RT: 3.55 - 5.50 SM: 7G



NL: 7.12E5
m/z=
174.14796-174.14970 F:
FTMS + p ESI Full ms2
174.15@hcd30.00
[50.00-375.00] MS
kb_140730164717

NL: 8.64E4
m/z=
174.14796-174.14970 F:
FTMS + p ESI Full ms2
174.15@hcd30.00
[50.00-375.00] MS
std-20ppt

Figure S3 Chromatograms of blank urine (a) and muscarine standard (RT: 4.56 min) at the concentration of 0.02 µg/L (b).