SUITABILITY TESTING BETWEEN COMMERCIAL SPE SORBENTS FOR THE SAMPLE CLEAN-UP IN STA USING LC-MS(/MS)

Tineke Decaestecker

E. Coopman, C. Van Peteghem, and J. Van Bocxlaer

Laboratory of Medical Biochemistry and Clinical Analysis,
Ghent University, Belgium

Outline

- Introduction and goal
  - ✓ DDA - fundamentals
- Study set-up
  - ✓ Flowchart
  - ✓ Extraction yield evaluation
  - ✓ Clean-up potential evaluation
- Results
  - ✓ Evaluated SPE-sorbents
  - ✓ Extraction yield evaluation
  - ✓ Clean-up potential evaluation
- Conclusion

Introduction & Goal

- **G**eneral **U**nknown **S**creening procedures

  ☝ no foreknowledge

  ⇨ **D**ata-**D**ependent **A**cquisition (DDA) ~ Q-TOF*

  * RCM 14, p. 1787-1792 (2000)

- Suitability test of SPE sorbents:

  ✓ Apolar
  ✓ Mixed-mode
  ✓ Polymeric

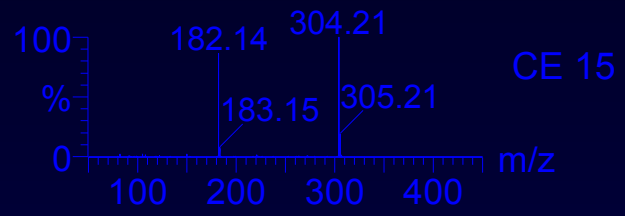
  } extraction yield ↔ cleanliness

  ⇨ Selection of best sorbent

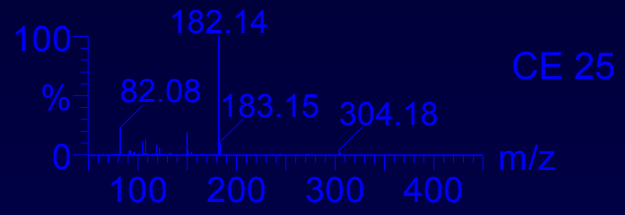
DDA – fundamentals (1)

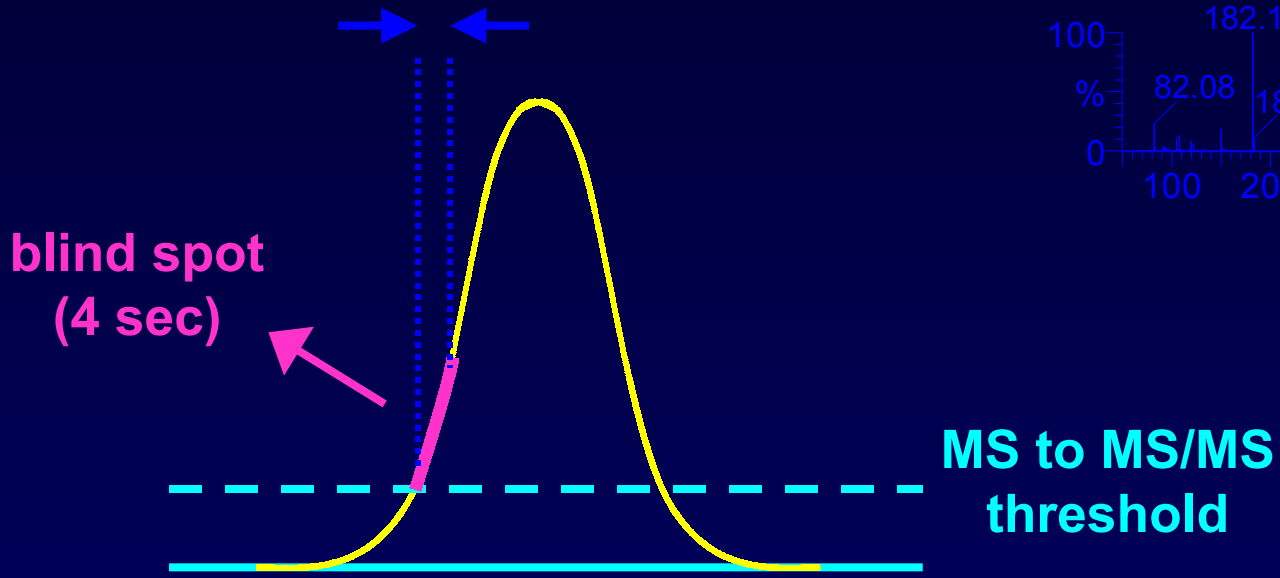
- Initially: QUAD = wide band-pass filter
  - precursor ion(s) > MS threshold
    - ⇨ switch to MS/MS
      - ° fragment ions ⇨ TOF
  - fragment ion(s) < MS/MS threshold
    - or total MSMS acquisition time
    - ⇨ switch back to MS

DDA – fundamentals (2)



Interruption for MS/MS } 2 spectra ≠ CE





DDA – fundamentals (3)

Qualitative + Quantitative

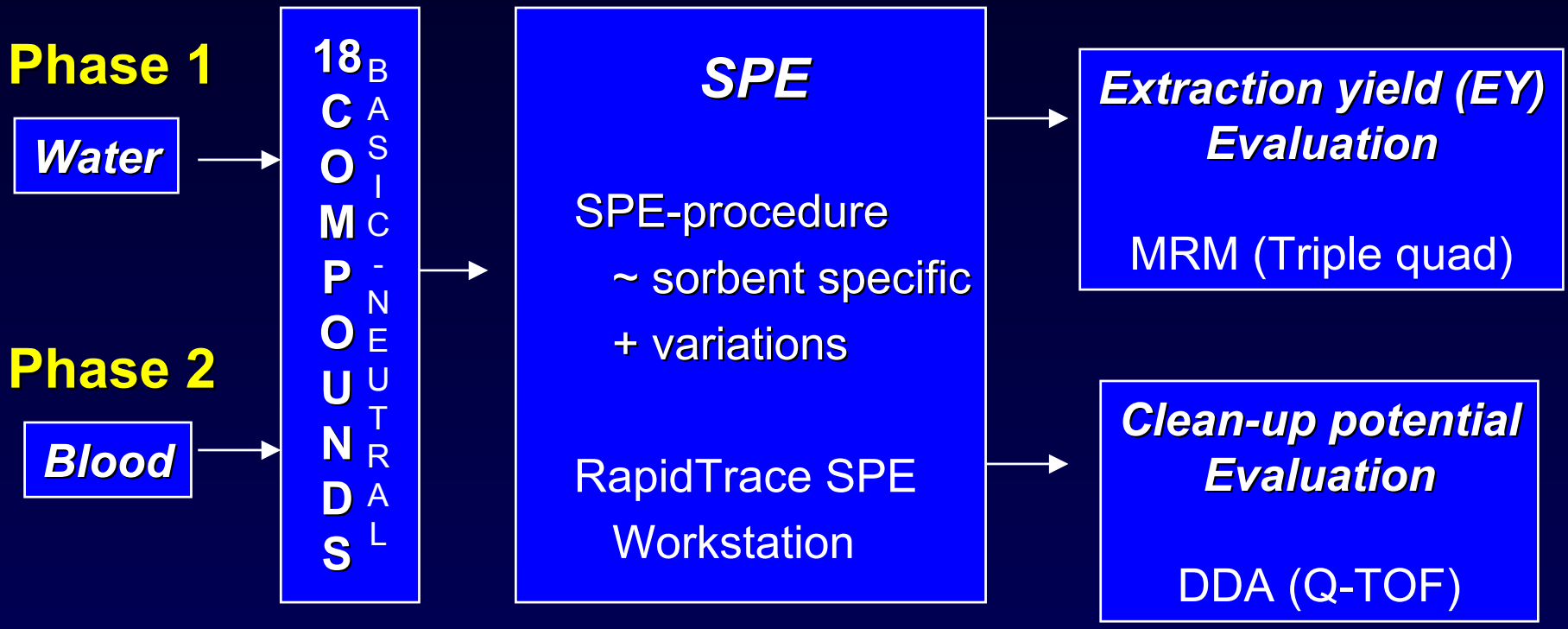
✓ MS spectrum ([M+H]$^+$)
✓ MS/MS spectra

✓ Integration of MS extracted ion chromatogram

Advantages DDA
- –No foreknowledge
- –Quantitative indication
- –Clean MS/MS spectra

} 1 run

Study set-up: flowchart



Extraction yield evaluation

For sorbent$_x$

	EY	
Compound 1	A%	Value 1 = F(A%)
Compound 2	B%	Value 2 = F(B%)
.	.	.
.	..	.
Compound 18	R%	Value 18 = F(R%)
		V=∑ Value 1..18

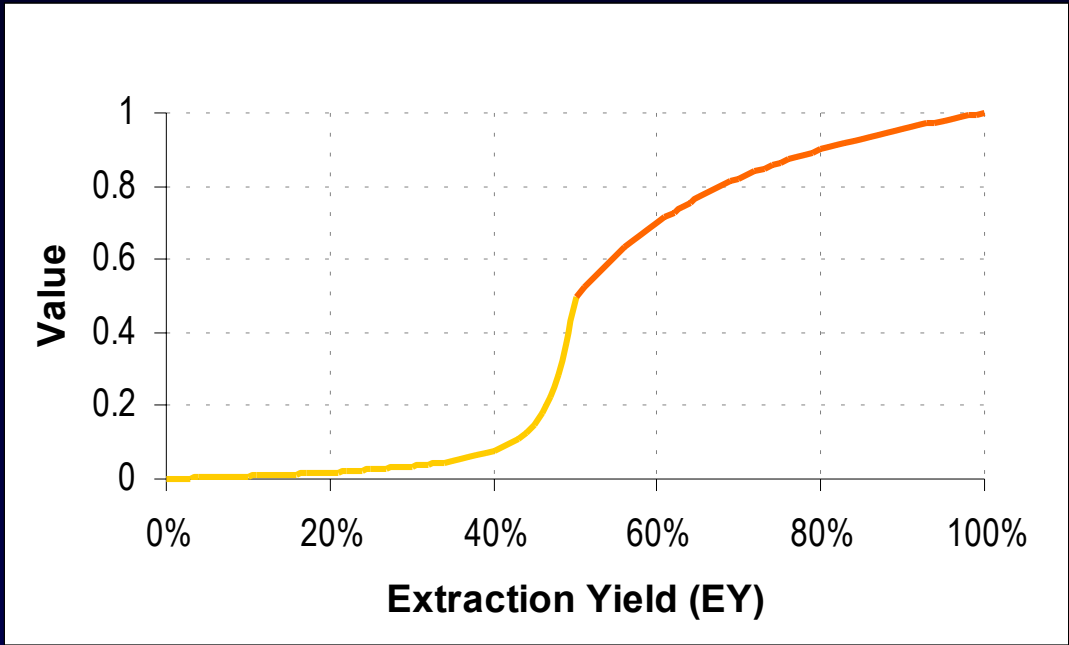
F() is a particular transformation function (see next slide).

Comparing the extraction yield of the sorbents

=

comparing the value V of the sorbents

Transformation function for extraction yield evaluation



Characteristics:

if Yield < 50%: more than linear penalty

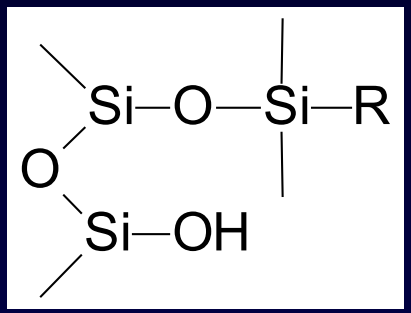
if Yield > 50%: more than linear reward

Clean-up potential evaluation

- 2 criteria:
  - ✓ DDA: number of spiked compounds “recovered”
  - ✓ CV% of EY

Results: 12 sorbents (3 categories)

APOLAR



MIXED-MODE

HCX: C8/SO_3^-
HCX3: C18/ SO_3^-
HCX5: C4/ SO_3^-

POLYMERIC

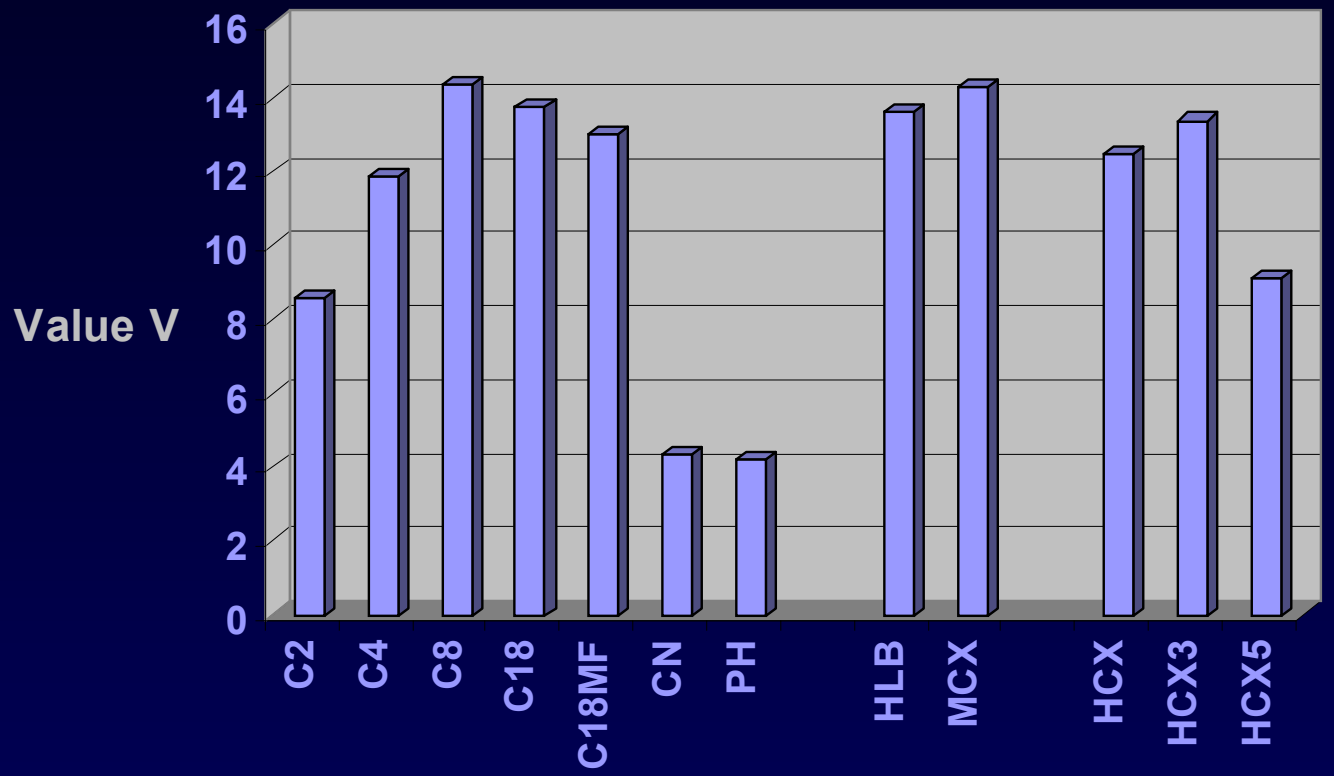
HLB: DVB-NP
MCX: DVB-NP/ SO_3^-

$R=(CH_2)_nCH_3$ C2, C4, C8, C18, C18MF

$R=C_6H_5$ PH

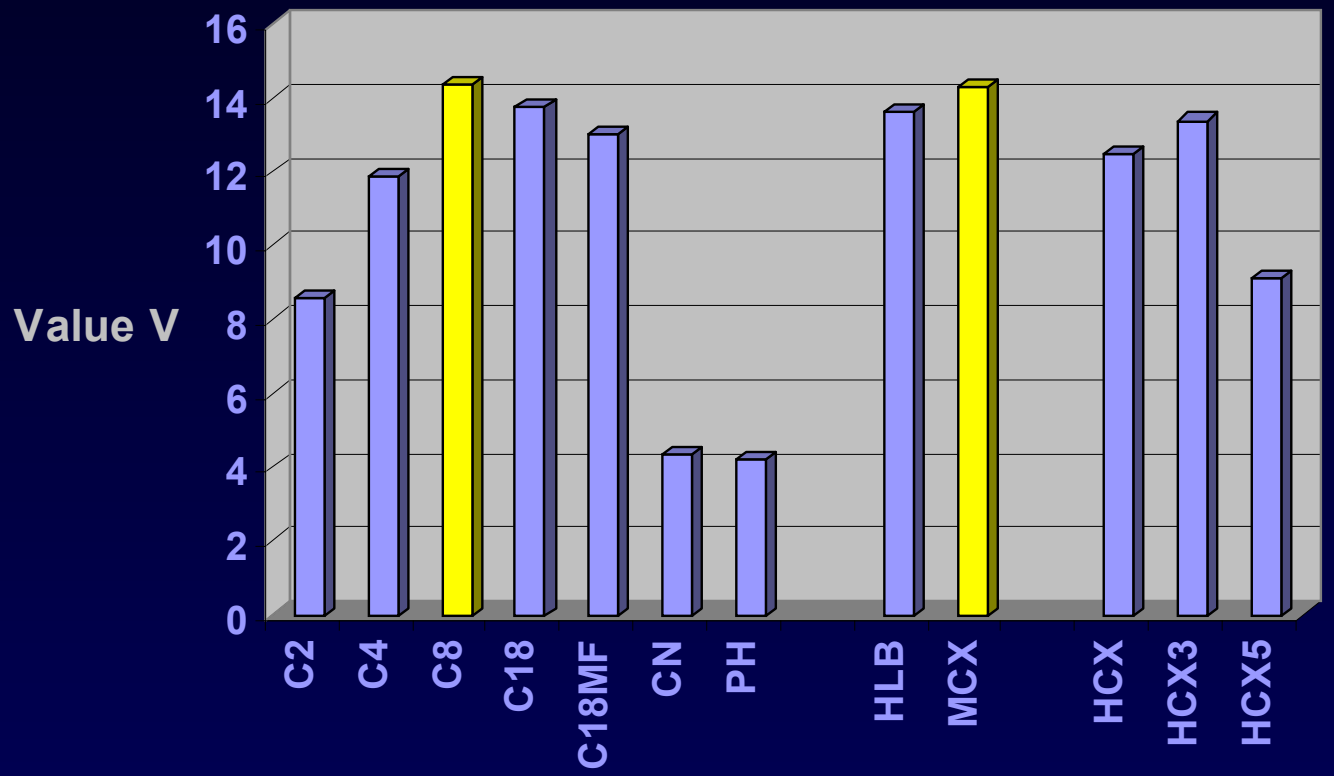
$R=(CH_2)_3CN$ CN

Results: extraction yield evaluation



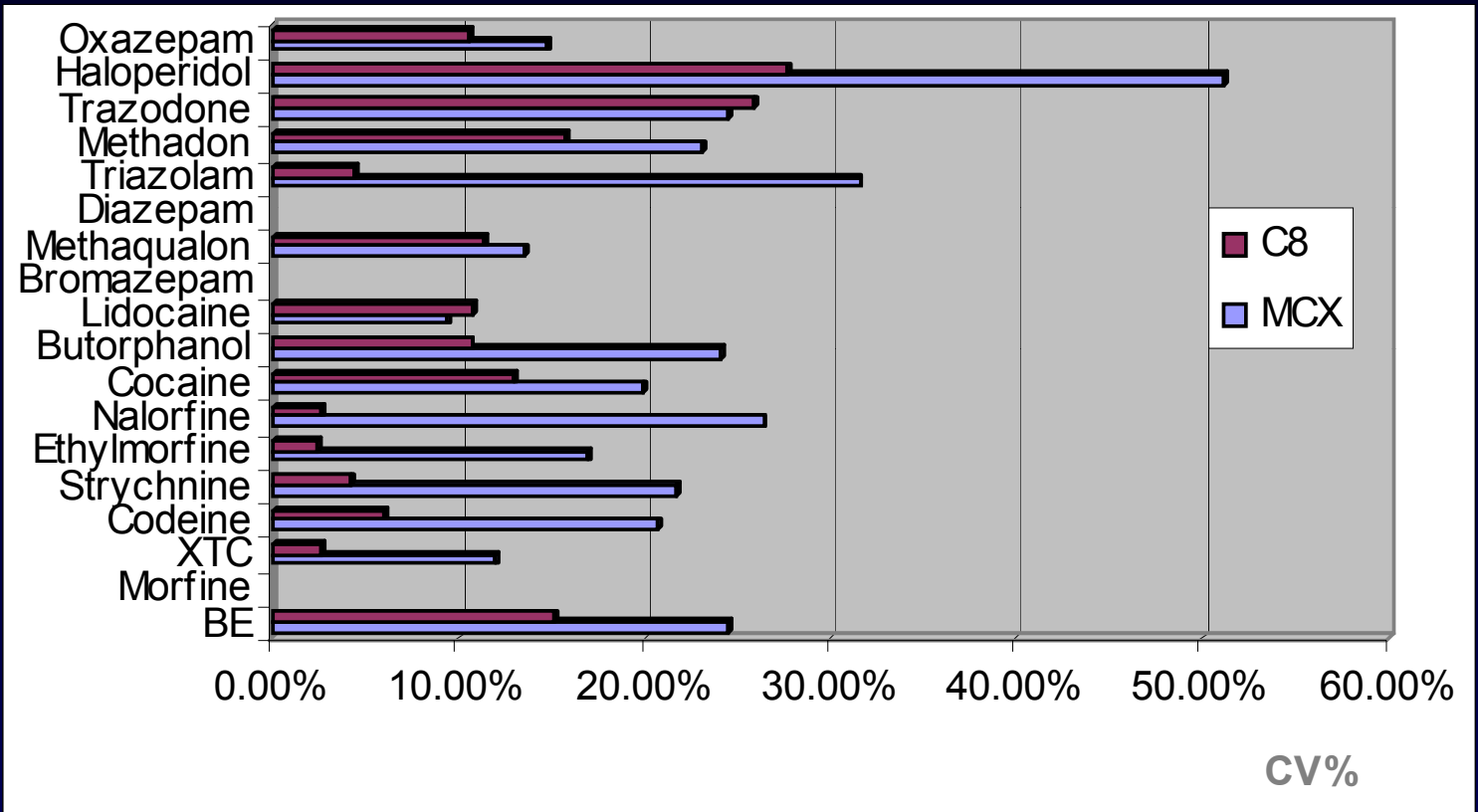
- Evaluation of 12 sorbents based on several extractions in triplicate

Results: extraction yield evaluation



- Evaluation of 12 sorbents based on several extractions in triplicate
- Choice of 2 sorbents for clean-up potential evaluation: C8 and MCX

Results: clean-up potential evaluation



- Fragmentation of 15 compounds
- Choice of sorbent: C8

Conclusions

- Evaluation of a range of SPE-sorbents on extraction yield and cleanliness criteria
- Selection of C8 for future use with DDA
- DDA: useful for STA