

Rapid Sample Preparation Protocols Using Micro Extraction by Packed Sorbent

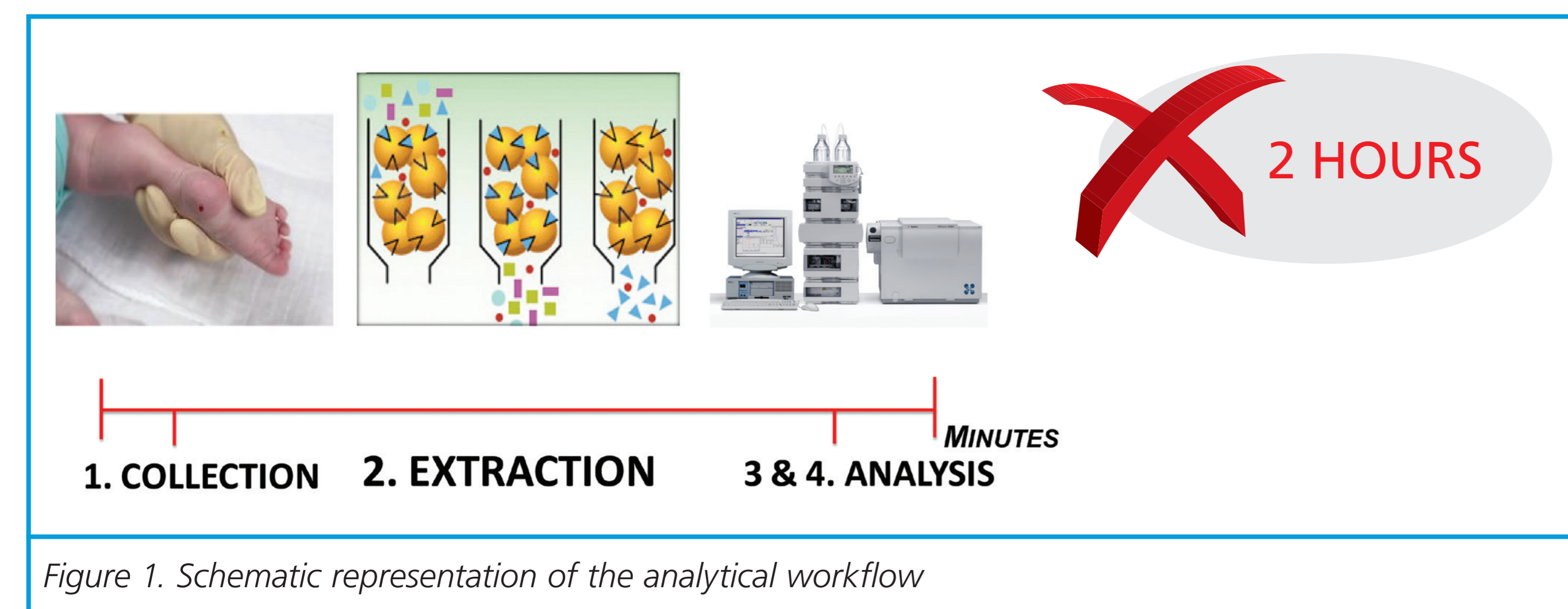
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Background

A typical analytical workflow is broken down into four areas (Fig. 1):

1. Sample Collection
2. Sample Preparation
3. Analysis
4. Data Processing

Sample preparation is **labor intensive** and **time consuming**, accounting for **80 % of the workflow**. It is also the primary area within the analytical workflow prone to error (Ref. 1) and much of the variation in the final result can be traced to the sample preparation. Hence, there is a need for more efficient sample preparation.



Micro Extraction by Packed Sorbent



Micro Extraction by Packed Sorbent (MEPS[®]) is a miniaturized version of Solid Phase Extraction (SPE) directly in the barrel of a syringe. Miniaturizing the solid support permits rapid workflows and requires only small volumes of sample and organic solvents (Fig. 2).

	Sample Volume	Time	Price	Solvents	Evaporation Step
SPE	4 droplets	15 min	High cost	4 droplets	Yes
MEPS	1 droplet	5 min	Low cost	1 droplet	No

Figure 2. Comparison Table SPE versus MEPS

At-Line Sample Preparation

- Incorporating a programmable digital analytical syringe for sample preparation dramatically reduces error.
- Sample preparation and injection protocols were developed using the single device streamlining workflows (Fig. 3 and Table 1).



Figure 3. eVo[®] MEPS: a programmable digital syringe with embedded SPE capability

Step	Aspirate (μL)	Dispense (μL)	Speed (μL/min)	Valve Position
Condition				
Methanol	50	50	600	1
Methanol	50	50	600	1
Equilibrate				
Water	50	50	600	1
Water	50	50	600	1
Sample load				
Urine (10 % v/v)	50	50	500	1
Urine (10 % v/v)	50	50	500	1
Wash				
Water	50	50	600	1
Water	50	50	600	1
Elute				
Methanol	50		600	2
Methanol		50	20	1

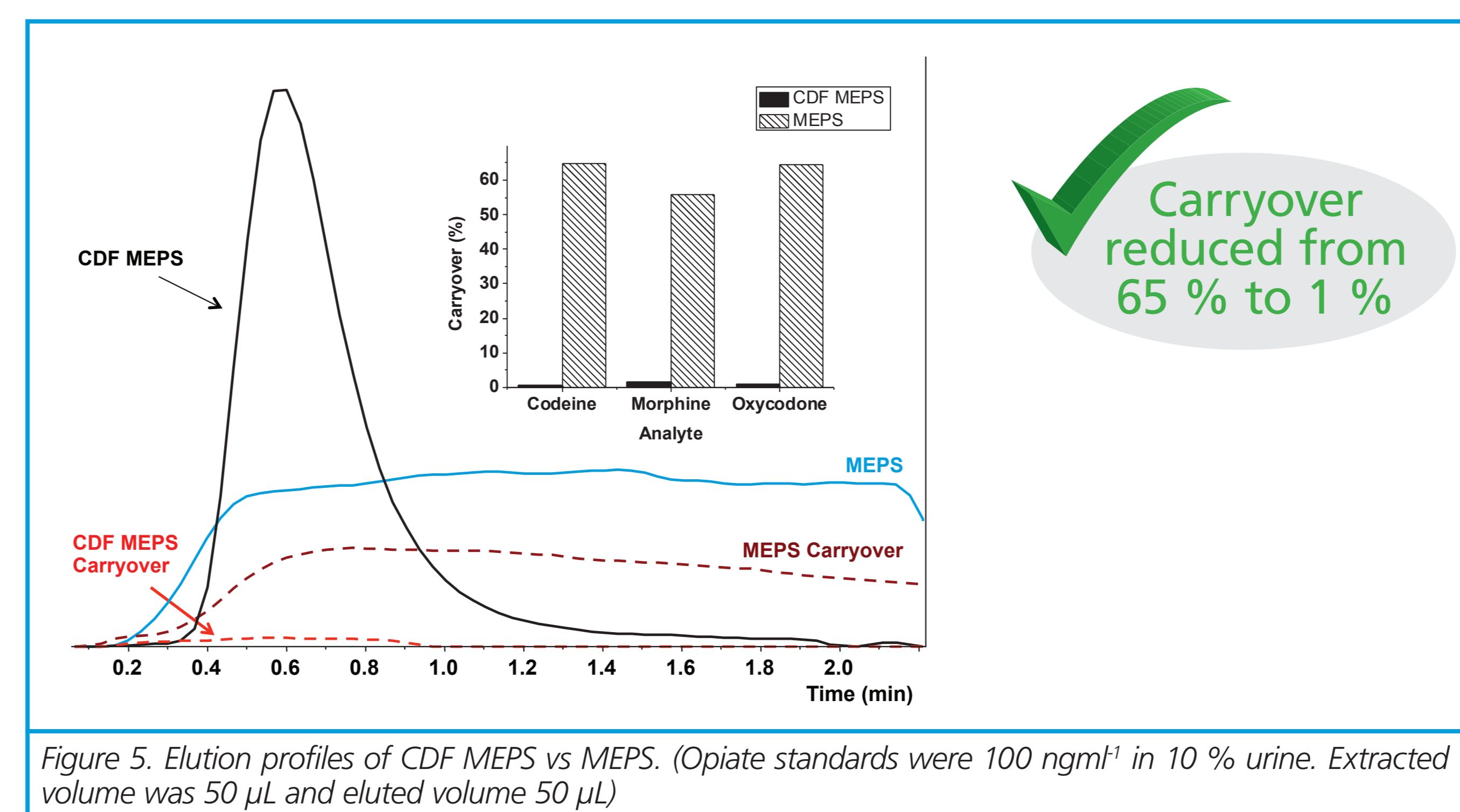
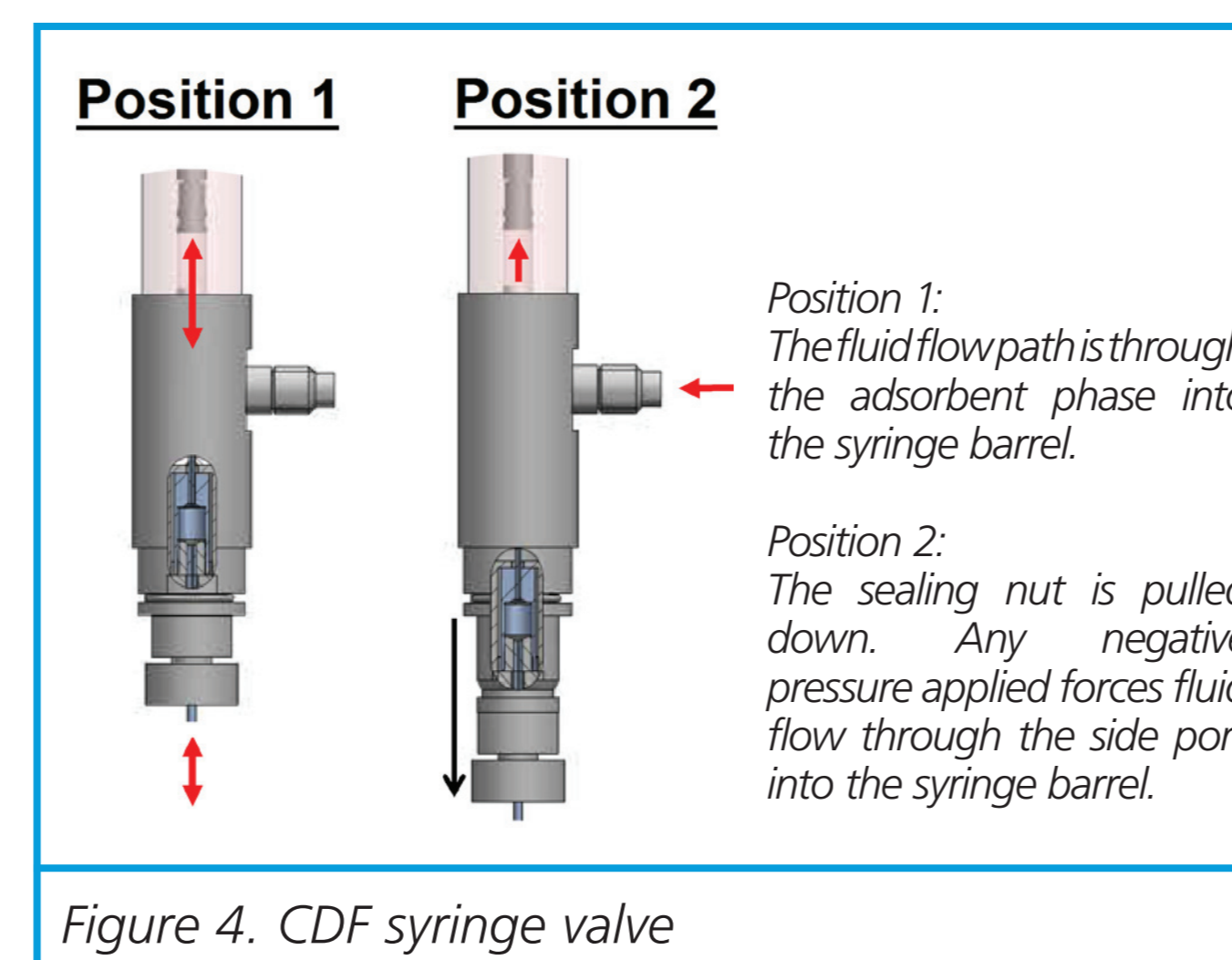
*Note: All solvents and samples contained 0.1 % formic acid

Table 1. eVo[®] MEPS programmed steps

Controlled Directional Flow

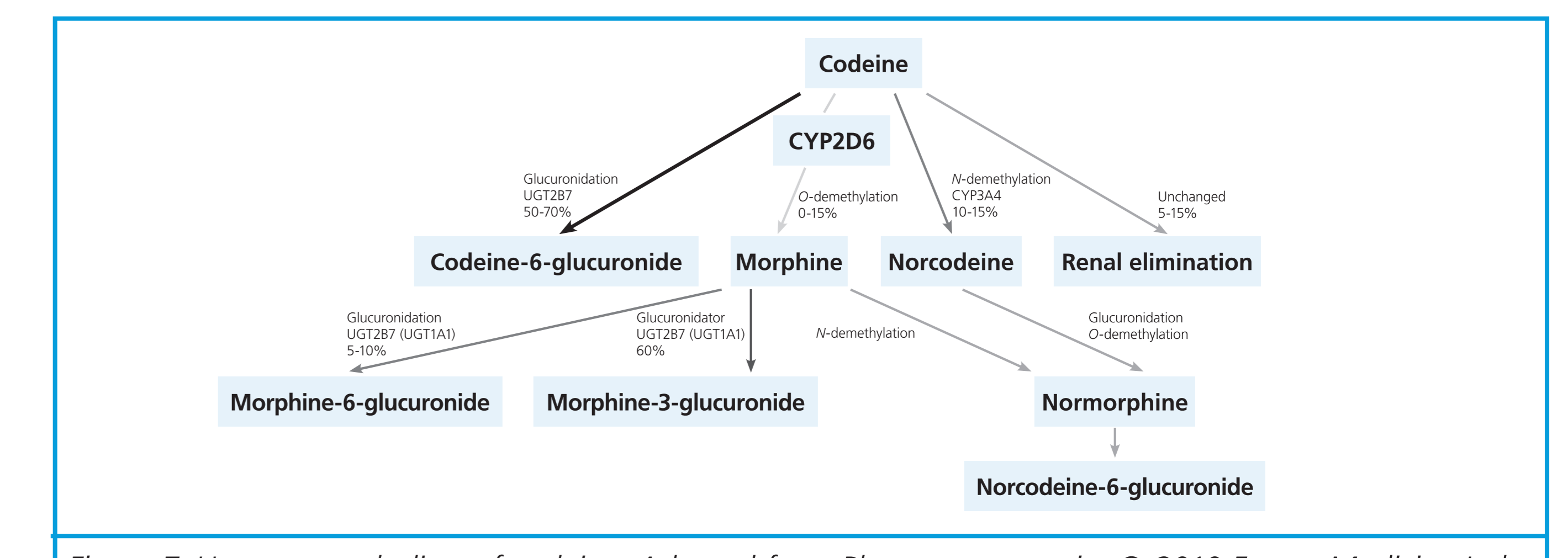
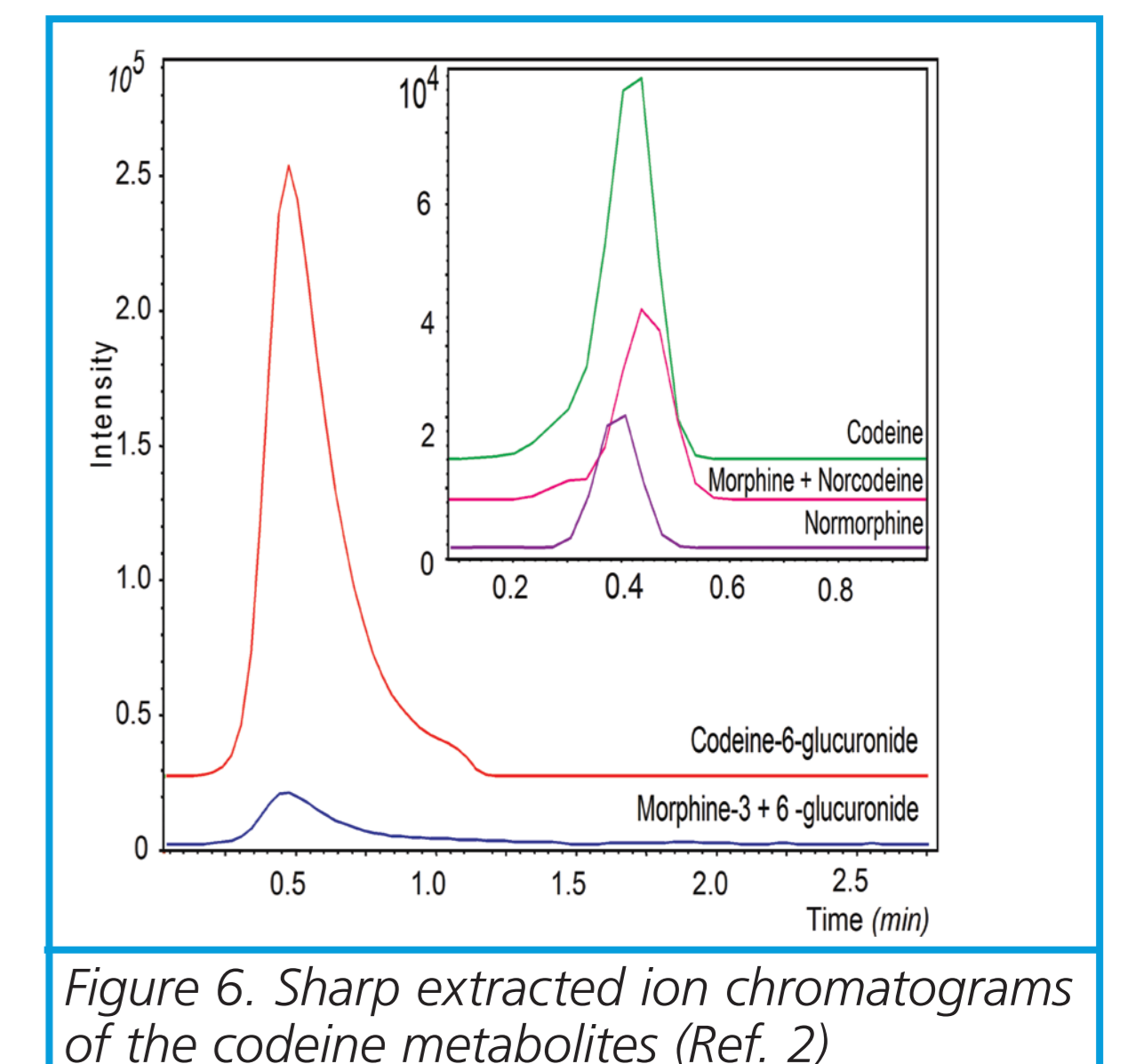
By introducing a two-way valve into the syringe barrel, the fluid flow path can be controlled (Fig. 4). In this way the elution solvent can be aspirated into the syringe bypassing the SPE bed minimizing the dilution effect seen with the traditional MEPS device. Controlled directional flow (CDF) MEPS delivers sharp, concentrated sample bands directly to the MS (Fig. 5), dramatically reducing carryover and eliminating the need to optimize elution protocols.

For sharp elution bands, aspirate eluant in position 2 and dispense in position 1.



Screening of Opiates and Metabolites from Urine

- Urine samples were prepared using MEPS workflow highlighted in Figure 3 and Table 1.
- Pure sharp concentrated sample bands were detected for all metabolites (Fig. 6 and 7).
- Opiate standards showed high recoveries, low detection limits, and good linearity. The CDF-MEPS workflow is robust (Table 2).



Analyte	Recovery (%)	Limit of Detection (ng/mL)	r ²	Matrix Effects (%)
Codeine	72	2	0.9986	32
Morphine	64	5	0.9994	37
Oxycodone	89	5	0.9981	42

Table 2. Opiate standards extracted from urine using MEPS

Conclusion

- Total analysis time of 5 minutes saves approximately 155 minutes.
- Significant reduction in sample carryover from 65 % to less than 1 %.
- A digital syringe provides control over the system leading to higher confidence in results.

Reference: [1] H. Kataoka, *Anal Sci* 27 (2011) 893. [2] Candish et al. *J. Sep Sci* (2012) 35, 2399-2406.

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