New features and benefits of automated sample preparation based on Robotic Technology

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Challenges related to sample preparation

Sample throughput / productivity

✓ Price per sample

✓ Batches analysis vs one by one analysis

✓ (Ultra-) trace analysis

Multiresidue

"all in one run" (e.g. pesticides in food, "mineral oil")

✓ Migration study



Conventional sample preparation techniques

- ✓ time consuming
- ✓ high consumption of reagents and samples
- ✓ high costs per analysis
- ✓ increased waste generation
- ✓ increase in human error



Sample preparation

- Dilution
- ✓ Mixing (vortexing, agitation)
- ✓ Heating
- Sonication
- Centrifugation and recovery of the sample
- Evaporation
- Preparation of the calibration solutions
- Liquid-liquid extractions
- ✓ Solid phase extractions
- ✓ etc



Conventional sample preparation techniques





Advantages of automated sample preparation

• Run 24/7 ("unattended" sample preparation &/or analysis)

- Reproducible timing (i.e. derivatisation before analysis)
- Higher throughput ("faster")

✓ In combination with fast analysis

- ✓ On-line versus off-line
- ✓ Low reagent consumption
- Extended application range -> several applications on one single work station
- Automated maintenance to reduce contamination



Sample prep automation – how to?

- Depends on the available modules
- Concentration ranges
- Possibility to miniaturize
- Possibility to use advanced injection and detection techniques



Configurations





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Method translation <-> miniaturisation

Reference method (ISO 28540)	Automated
1 L water + IS	15 mL + IS
Target LOD: 1 ng/L	Target LOD: 1 ng/L = 15 pg/15 mL
LLE with 25 mL hexane	μLLE with 3 mL hexane
100% recovery: 1 ng/25 mL	100% recovery: 15 pg/3 mL
Recover organic phase	Centrifuge (?)
Drying on Na ₂ SO ₄	Transfer 1 mL to 2 mL vial with Na ₂ SO ₄ 100% recovery: 5 pg/1 mL
Concentration to 1 mL	Vortex & centrifuge (?)
Final concentration: 1 ng/mL = pg/µL	Final concentration: 5 pg/mL = fg/µL
GC-MS analysis	GC-MS/MS analysis
1 μL injection	40 μL injection (LVI-PTV)
Amount on-column: 1 pg	Amount on-column: 0.2 pg
MS → MS/MS	



Automation – Maestro scheduler



- User-friendly software
- Prep-ahead option
- Allows to automatically dispense into various types of destinations with or without moving the destination
- For online configuration -> "just in time" sample preparation (i.e. derivatisation)
- Possibility to run offline





Automation – Maestro scheduler

W Sample Prep

Prep. Settings Vial Range 1 Prep. Action Settings Syringe: 2.5mlALX a ADD MPS Right MPS ~ Action ~ ~ Method add water meth 1 Edit New Delete Description Syringe: 2.5mIALX SFSWash4 Vial AUTO 🗸 Source ~ Destination Tray2,VT32-20 ~ Vial AUTO ~ He Insert 2. Replace × Delete 🔀 Clean Add Action MPS Method / Value Source Vial Destination Vial ~ ADD Right MPS add water meth 1 SFSWash4 Tray2,VT32-20 Right MPS Tray2, VT32-20 mVorx,mVTC1-10 U MIX Left MPS mix poudre MOVE Right MPS mVorx,mVTC1-10 Tray2,VT32-20 ADD ADD Tray2,VT32-20 Tray2,VT32-20 **Right MPS** add ISTD C11Me / C13TG methode 1 25 Right MPS add ISTD C11Me / C13TG methode 1b Trav2, VT32-20 Trav2.VT32-20 25 Tray2,VT32-20 MOVE Right MPS mVorx,mVTC1-10 U MIX Left MPS mix ISTD MOVE Right MPS Trav2.VT32-20 mVorx,mVTC1-10 ADD ADD MOVE Right MPS add NaOCH3 5% Wash2 Tray2,VT32-20 Right MPS add NaOCH3 5% b Wash2 Tray2,VT32-20 Right MPS Tray2, VT32-20 mVorx,mVTC1-10 U MIX Left MPS NaOCH3 mix mVorx,mVTC1-10 MOVE Right MPS Tray2,VT32-20 HAN CO Right MPS wait NaOCH3 Right MPS add hexane SESWash3 Tray2, VT32-20 **Right MPS** add sol de neutr Wash4 Tray2,VT32-20 Right MPS add sol de neutr Wash4 Tray2, VT32-20 Tray2,VT32-20 **Right MPS** add sol de neutr Wash4 Right MPS Trav2.VT32-20 add sol de neutr Wash4 Tray2,VT32-20 **Right MPS** mVorx,mVTC1-10 U MIX Left MPS mix after neutr MOVE Right MPS mVorx,mVTC1-10 Centrifa.CT6-10 1 MOVE Right MPS Tray2, VT32-20 29 Centrifg,CT6-10 4 Right MPS CF200 centrifuge MOVE **Right MPS** Centrifg,CT6-10 4 Tray2,VT32-20 29 MOVE Centrifg,CT6-10 Tray2, VT32-20 Right MPS 1 MOVE Tray1.VT98 Tray2.VT32-20 Left MPS 8 ADD Right MPS transfer to 2 mL (dry) meth 1 poudre Trav2.VT32-20 Trav2.VT32-20 8 Left MPS Tray2,VT32-20 8 mVorx,mVTC8-2 U MIX Left MPS Dry mix A MOVE Left MPS mVorx.mVTC8-2 Trav1.VT98 WAIT Left MPS wait before inj Left MPS post injection delay = 0.5 min ADD Right MPS rinse acetone SFSWash1 SFSWaste ---------OK Cancel Help



Typical application

✓ Life science

- ✓ FAME's analysis
- Organic acids
- ✓ Food analysis
 - ✓ FAME's
- Cosmetics analysis

Environmental

- \checkmark µLLE for pesticides & PAH's
- ✓ VOC's in water



Conclusions

- Take advantage of state-of-the-art instrumentation
- Miniaturized automated reduced solvent consumption – increased productivity – safer
- But...
 - Sampling must be reconsidered in function of (automated) sample prep
 - There is a minimum sample amount required for reliable analysis (sample homogeneity)
 - New methods should be compared to existing ones and properly validated (certified materials, proficiency tests, demonstrate equivalence)





