Analysis of 1,4-Dioxane in Drinking Water with Semi-Automated Solid Phase Extraction Using EPA Method 522

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- 1,4 dioxane found in drinking water
- In US regulated by EPA method 522
- Demand for fast, reliable and reproducible laboratory analysis

- Solvent for paper, cotton and textile processing
- Manufacturing chemical industry
- Coolant in automobiles

- Allergic symptoms – eyes, nose
- Kidney and liver damage
- Suspected carcinogenic
- Miscarriage/stillbirth
- Transfer via breast milk

- Many labs analyze drinking water samples
- Liquid-Liquid Extraction (LLE) or Solid Phase Extraction (SPE) can be used
- In both cases organics are transferred from water sample to an organic solvent
- With SPE compounds are first deposited on cartridge or disk, then eluted

LLE/CLE	Semi-Automated SPE
Open to laboratory background	Closed system
Uses >360 mL solvent	Uses < 60 mL solvent
Shaking / Continuous process	Filtration process
Forms emulsions requiring centrifuging	No emulsions formed
Little Selectivity	Wide Selectivity (adsorbent)
Requires water removal	In-line water removal

LLE/CLE	Semi-Automated SPE
No Separation of waste	Separates Aqueous and OrganicWaste
More volume to evaporate	< 60 mL solvent to evaporate
High solvent emission	6 times lower solvent emission
CLE uses a lot of solvent	Easily Capture Solvent
Requires lots of solvent for cleaning	Lower solvent and solvent disposal costs


360 ml extract
from manual
extraction


<60ml
extract from
FMS
PowerPrep
SPE

- Semi-automated SPE done by many labs around the world
- Cheaper than fully automated systems
- Important that system is reliable and fast
- Should be able to use variety of cartridges

FMS

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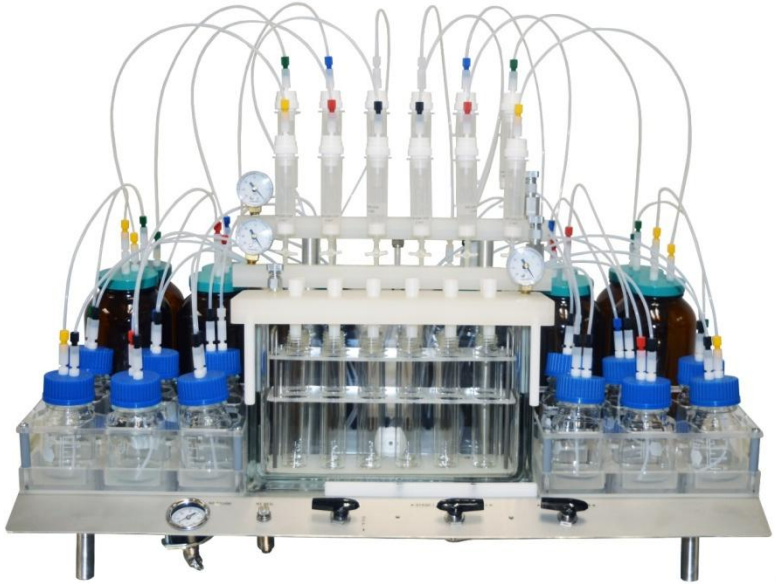
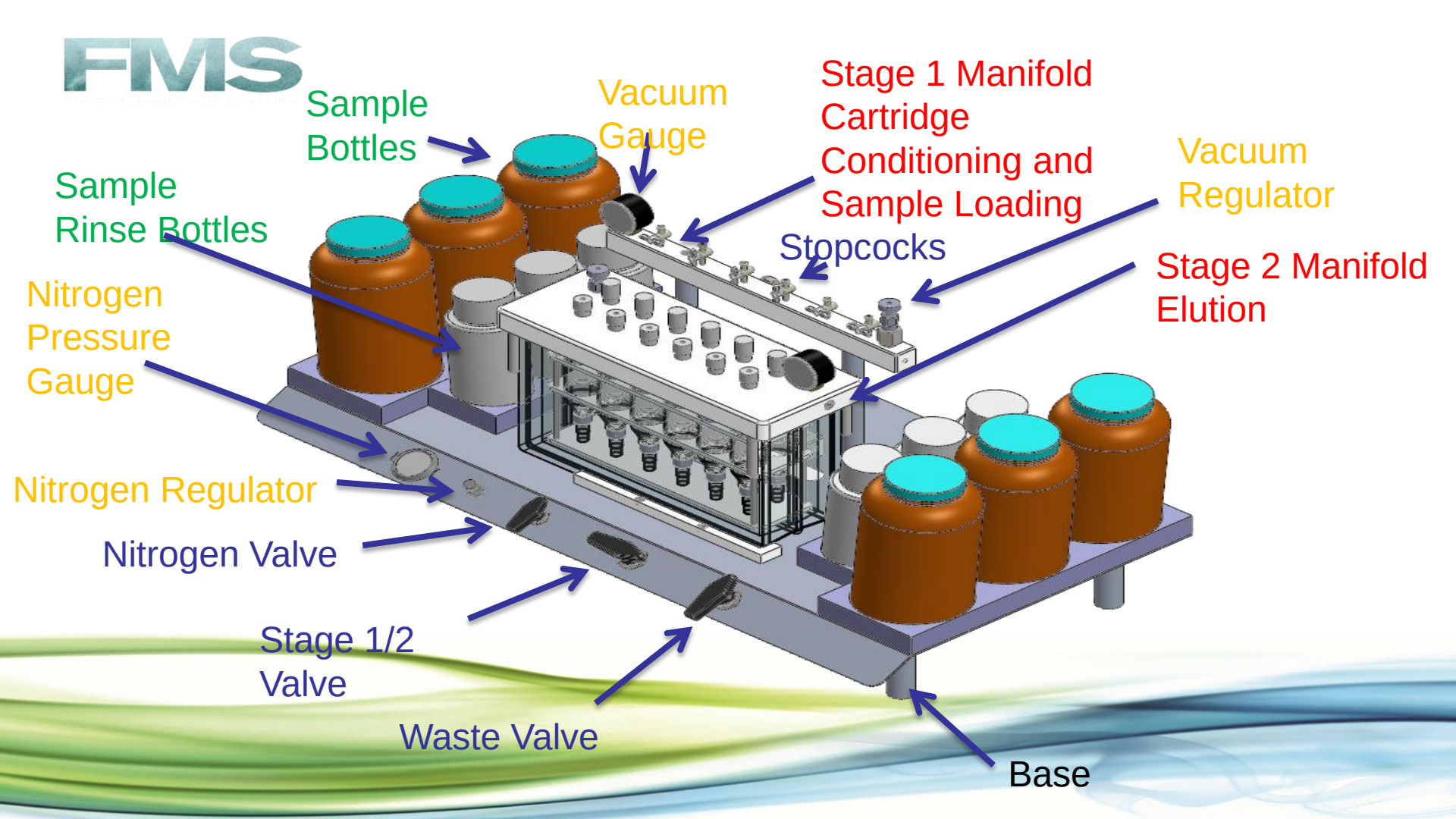
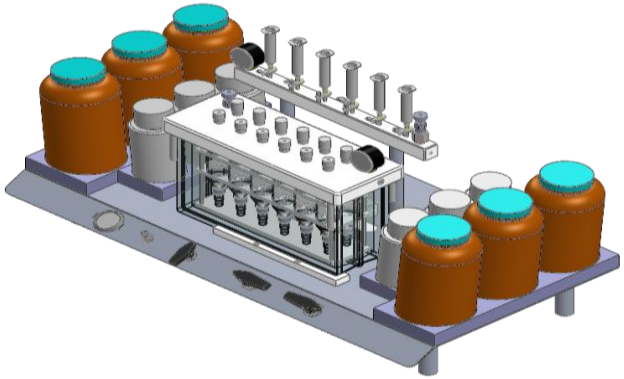



FMS
Sample Bottles
Vacuum Gauge
Stage 1 Manifold Cartridge Conditioning and Sample Loading
Vacuum Regulator
Sample Rinse Bottles
Stopcocks
Stage 2 Manifold Elution
Nitrogen Pressure Gauge
Nitrogen Regulator
Nitrogen Valve
Stage 1/2 Valve
Waste Valve
Base

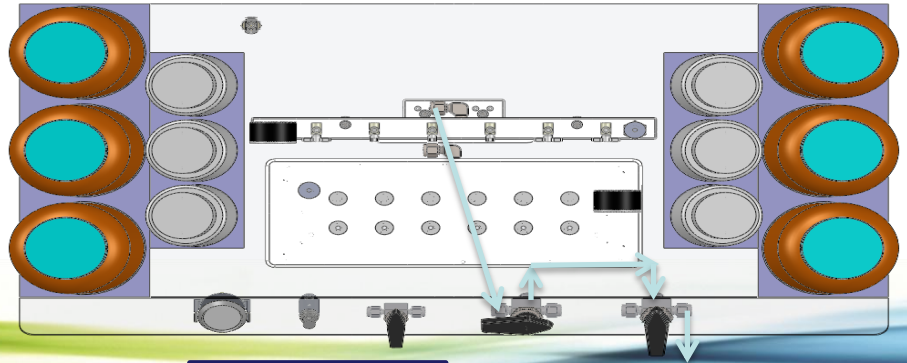


Flow Path

Nitrogen Valve

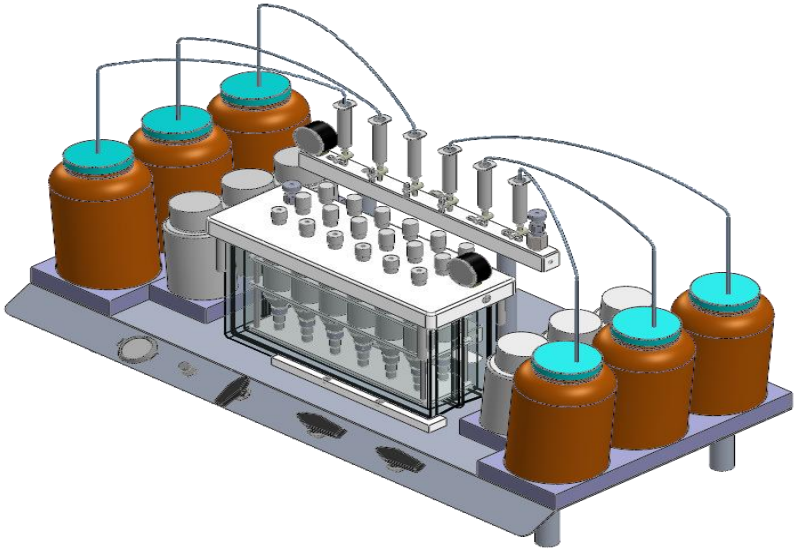
Stage 1/2 Valve

Waste Valve



Vacuum Pump

OW



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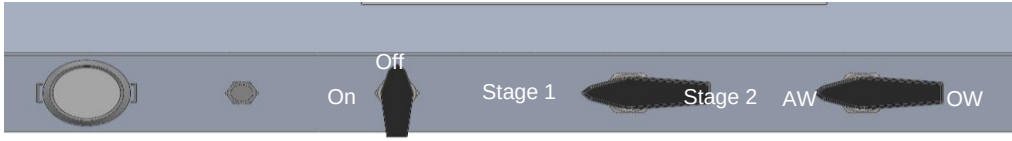

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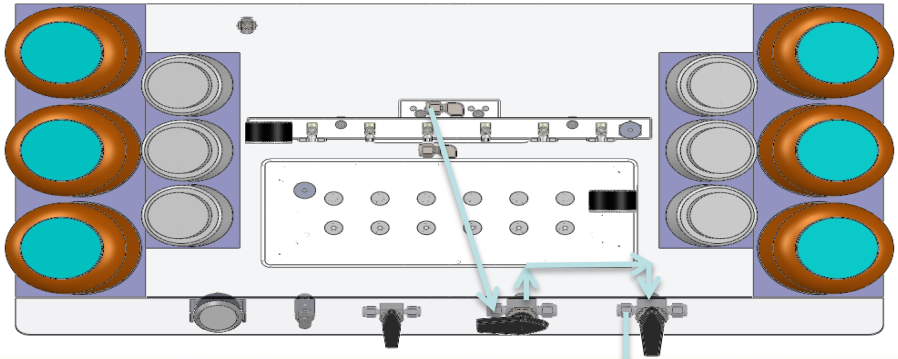

Vacuum Pump

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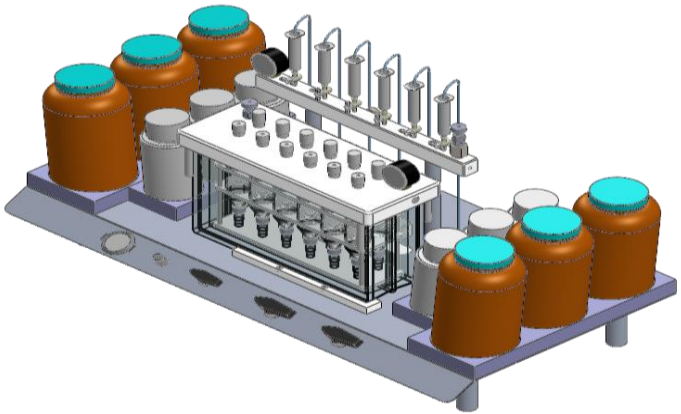

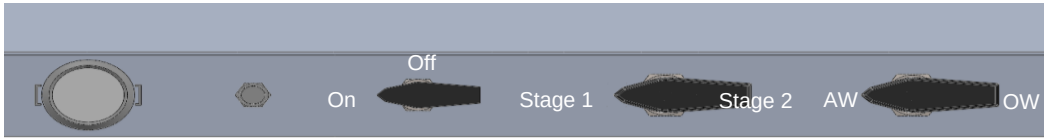

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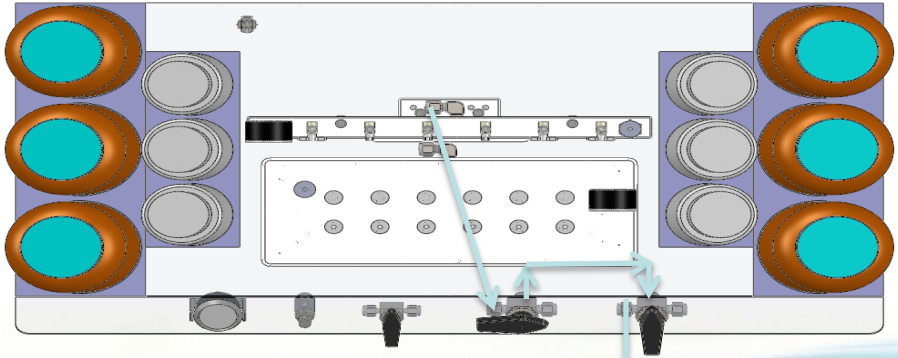

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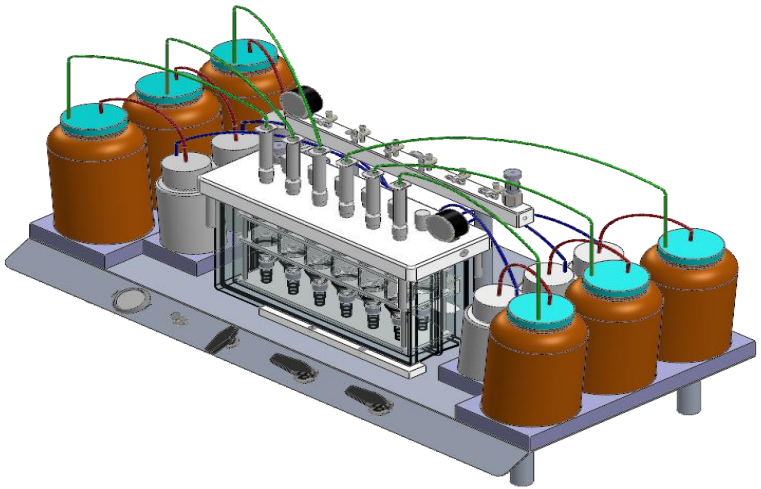

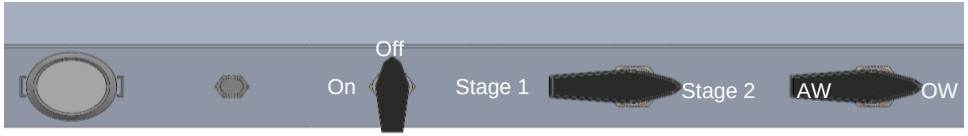

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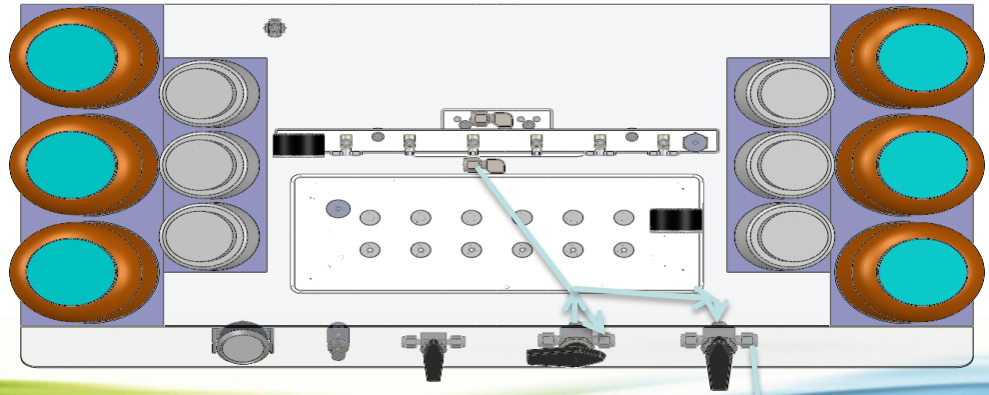

Flow Path

Vacuum Pump

OW

- Simple to Operate No Computer or Electronics
- Fast Runs 6 Samples in 20 ~ 50 min (depending on sample size)
- High Throughput Runs 6 Samples in Parallel
- Flexible Uses All SPE Cartridge Sizes
- Semi Automated Vacuum Sample Loading & Valve Selection for Separating Aqueous and Organic Waste

- Quality Consumables: Guaranteed Certified Cartridges
- Bottle Rinse: Automated Bottle Rinse
- In-line Extract Drying
- Reliable No Maintenance Required
- Zero Cross-Contamination, No Shared Tubing & Fittings

- 12 samples (100 or 500 mL water each) are prepared and acidified with sodium bisulfate till pH ~ 4
- Spike with 522 standards
- Put sample bottles in place and fill rinse bottles with 9 mL dichloromethane
- FMS 2 g coconut charcoal cartridges are installed in each of the positions.

Stage 1:

- Vacuum is turned on
- Cartridges are conditioned with 3 mL dichloromethane, 3 mL methanol, 3 mL methanol (keep wet) and 6 x 3 mL water (keep wet)
- Samples are loaded across cartridges under vacuum at 10 mL/min
- Cartridges are dried with vacuum for 10 min
- Sample bottles are automatically rinsed from the rinse bottles with 9 mL dichloromethane

Stage 2:

- Small amount of dichloromethane is pulled through cartridges, soak 1 min
- Rest of dichloromethane is pulled from the sample bottles thru the cartridges and collected
- In line sodium sulfate drying

Direct-to-Vial

- 6 (250mL) and 12 (50mL) position models for extractions, direct-to-vial connections
- Dry bath heating element
- Independent secondary heater for extract nipple (can be disabled)
- Sensor controlled
- Pre-heat 20 min at 35 oC, 35 oC sensor mode, 5 psi nitrogen
- Savable temperature log capability.

- Samples reduced to 1 mL under nitrogen flow
- Samples analyzed in 1 mL DCM
- Semi-Volatiles analyzed with low resolution GC/MS (SIM)

	1,4-dioxane-d8 recovery (%)	1,4-dioxane conc (ug/L)	STDEV
Run # 1	89.4	1.03	
Run # 2	85.0	0.98	0.06
Run # 3	82.0	1.01	

- EZSpe delivers excellent recoveries for 1,4-dioxane within 80-120 % method window
- Runs 12 samples in parallel
- Gets data in under 2h
- No maintenance required
- No separate water removal step needed (in-line drying)
- Other applications are beverages, milk and serum