

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

Ruud Addink and Tom Hall  
Fluid Management Systems  
Billerica MA



- 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**

Open to laboratory background

Uses >360 mL solvent

Shaking / Continuous process

Forms emulsions requiring centrifuging

Little Selectivity

Requires water removal

## **Semi-Automated SPE**

Closed system

Uses < 60 mL solvent

Filtration process

No emulsions formed

Wide Selectivity (adsorbent)

In-line water removal

## **LLE/CLE**

No Separation of waste

More volume to evaporate

High solvent emission

CLE uses a lot of solvent

Requires lots of solvent for cleaning

## **Semi-Automated SPE**

Separates Aqueous and Organic Waste

< 60 mL solvent to evaporate

6 times lower solvent emission

Easily Capture Solvent

Lower solvent and solvent disposal costs

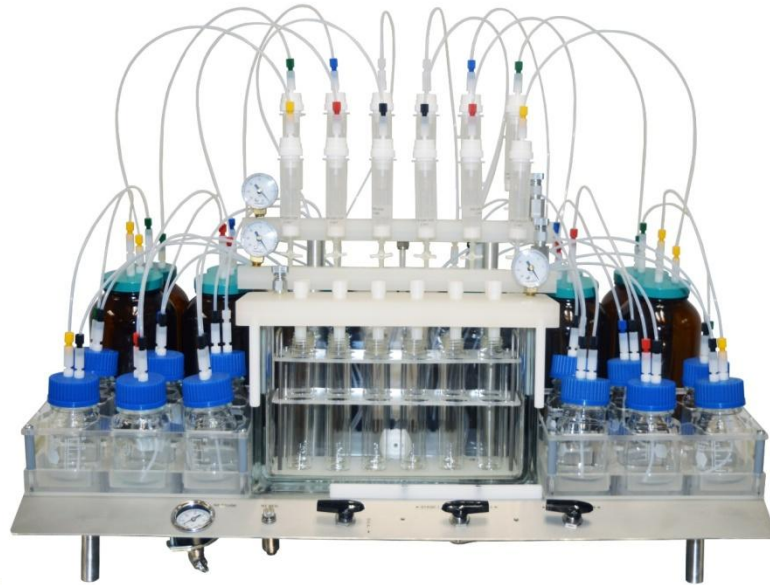


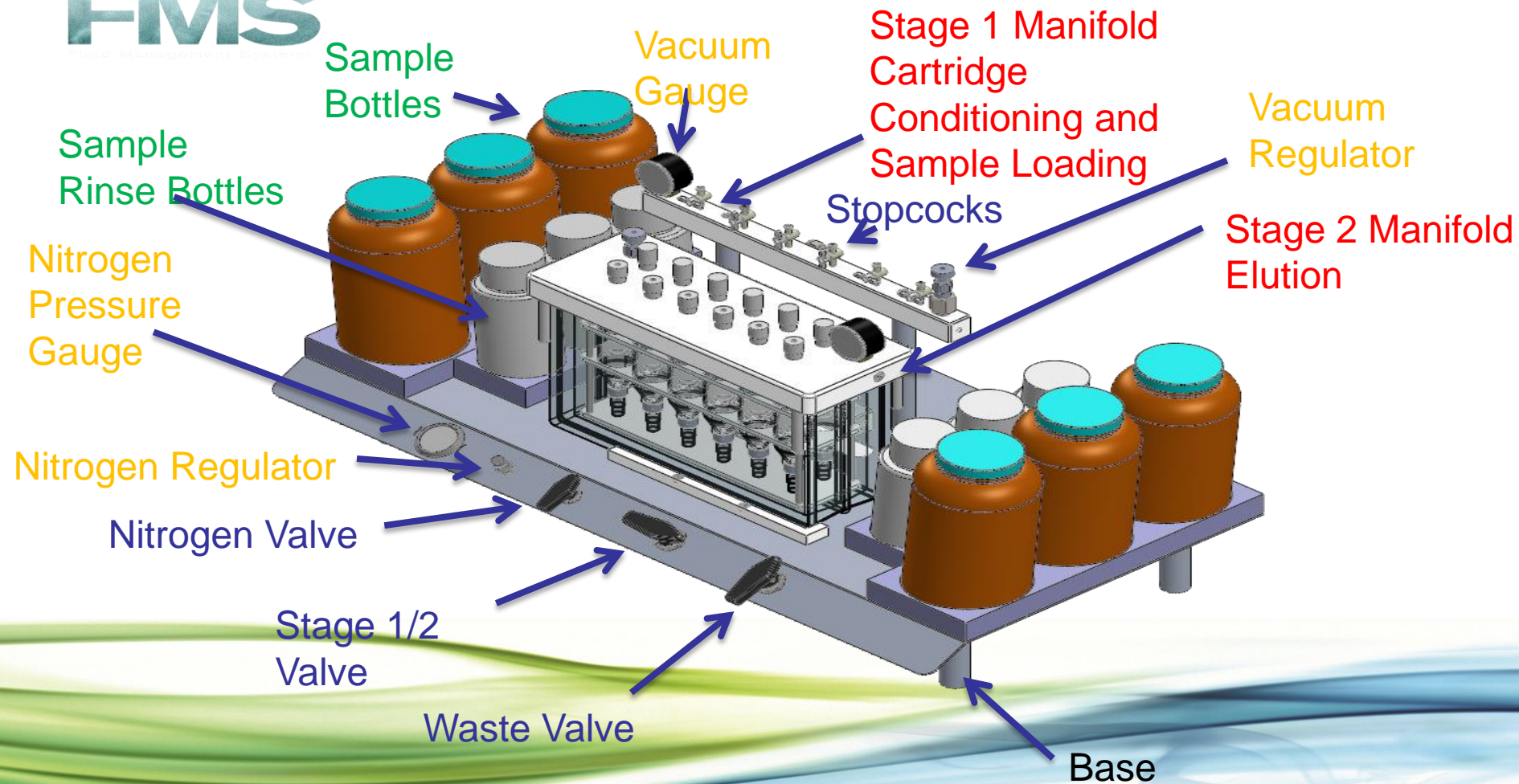


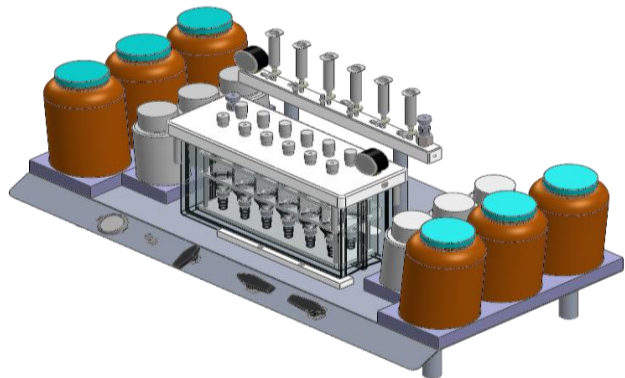


- Semi-automated SPE done by many labs around the world
- Cheaper than fully automated systems
- Important that system is reliable and fast
- Should be able to use variety of cartridges









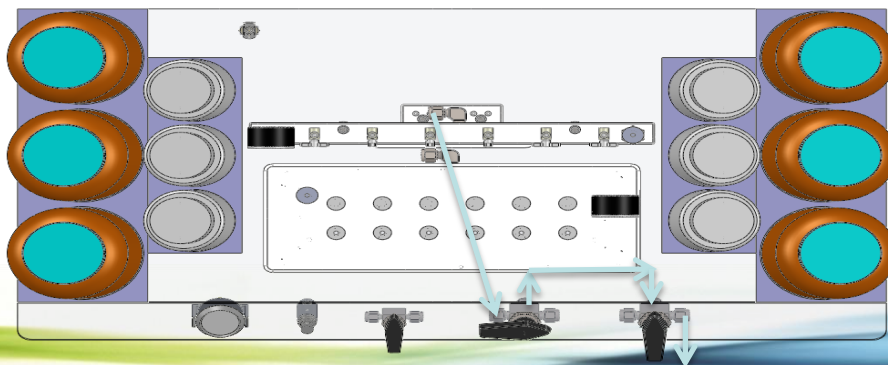
Flow  
Path



Nitrogen  
Valve

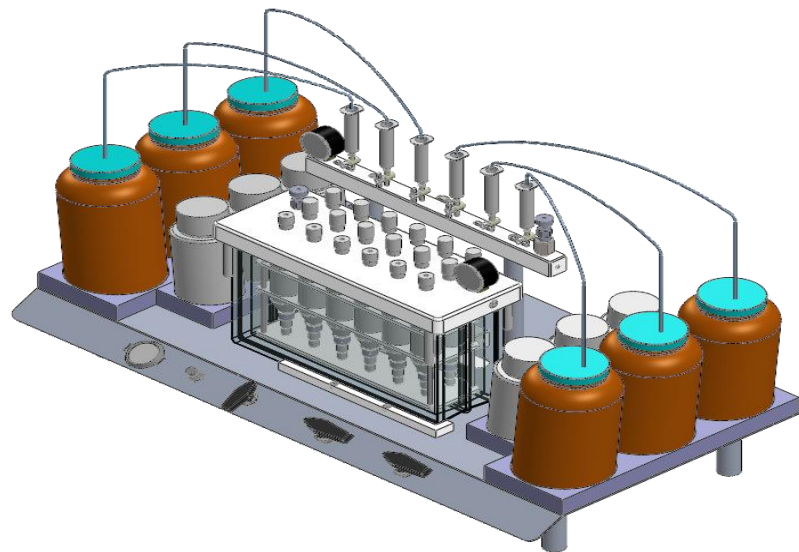
Stage  
1/2  
Valve

Waste  
Valve

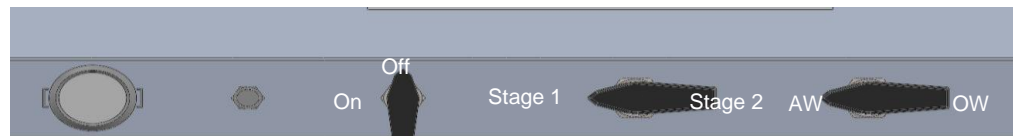


Vacuum  
Pump

OW



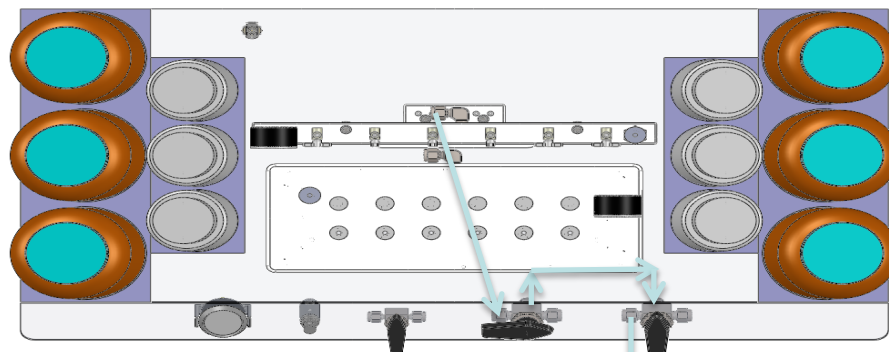
Flow  
Path



Nitrogen  
Valve

Stage  
1/2  
Valve

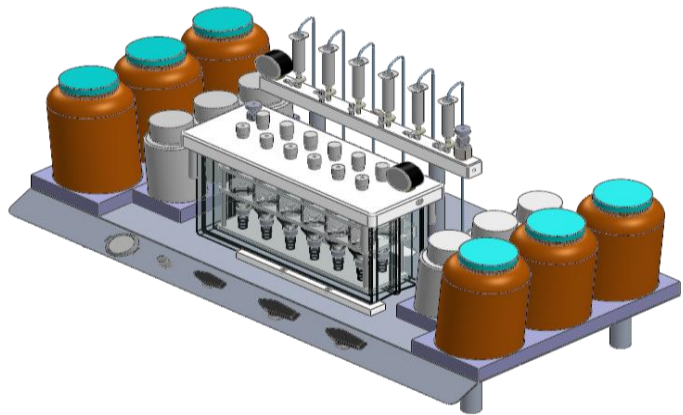
Waste  
Valve



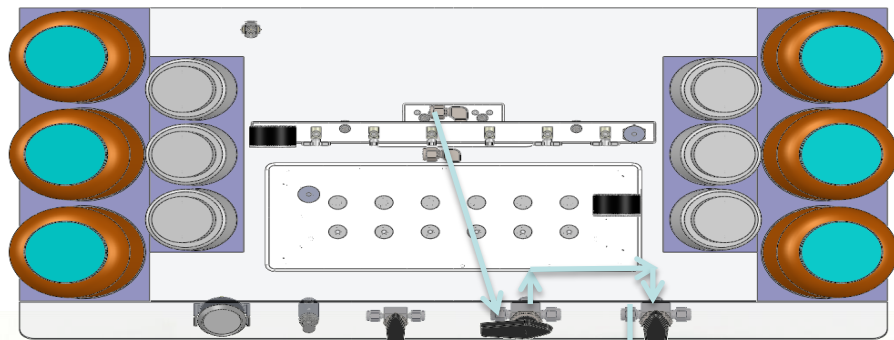
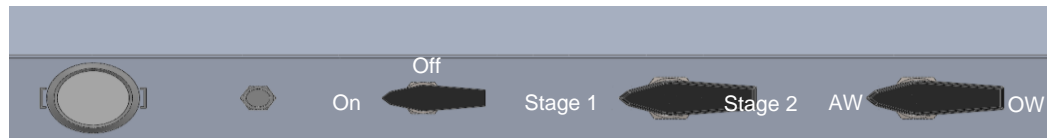
Vacuum  
Pump

AW



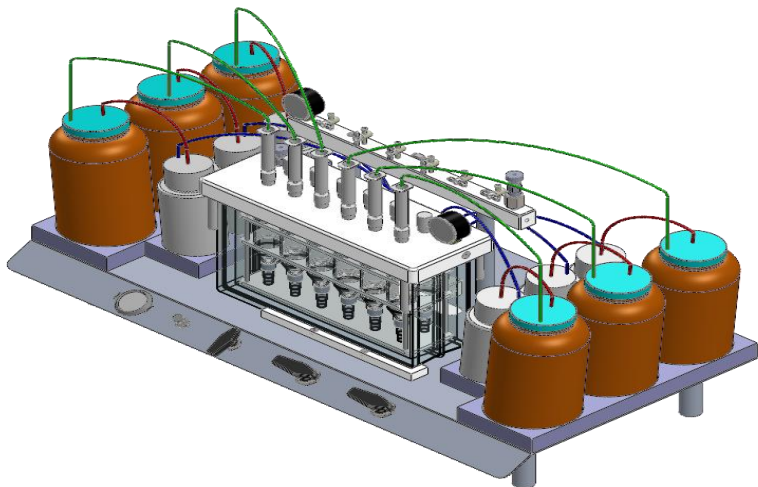


Flow  
Path

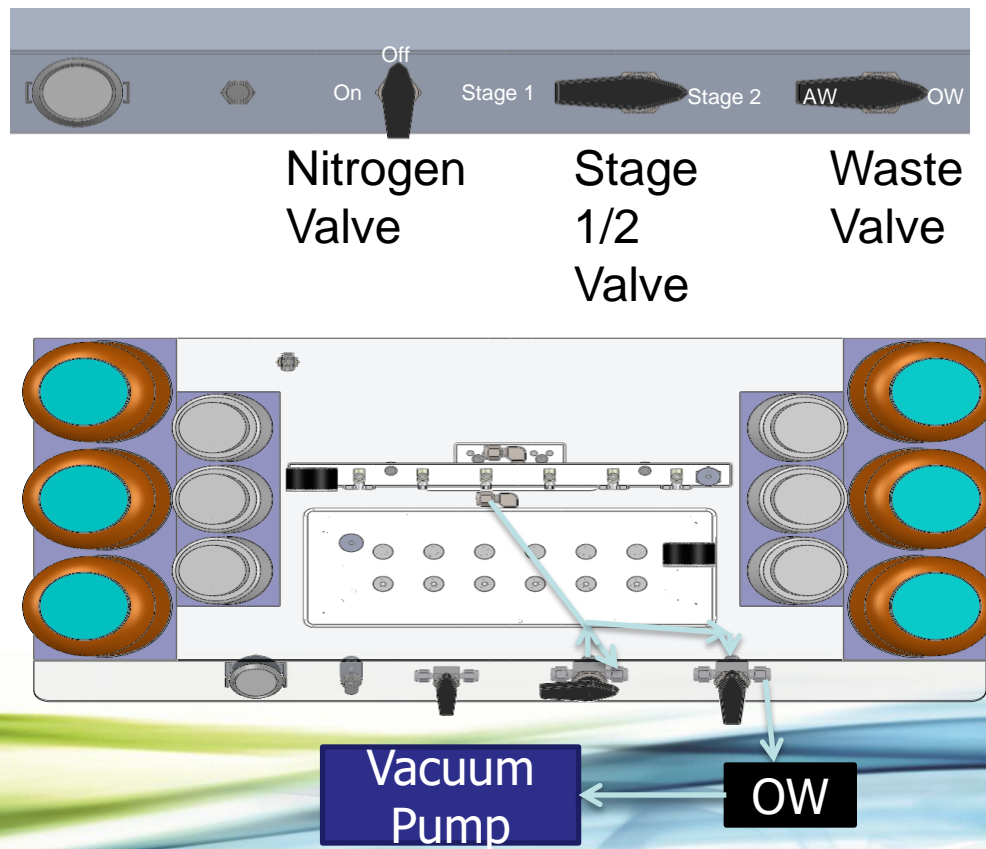


Vacuum  
Pump

← AW



Flow  
Path



- 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  $\sim 4$
- 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)



	<b>1,4-dioxane-d8</b>	<b>1,4-dioxane</b>	<b>STDEV</b>
	<b>recovery (%)</b>	<b>conc (ug/L)</b>	
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





Fluid Management Systems, Inc.

900 Technology Park Drive

Billerica MA 01821

Phone 617-393-2396

Website: [www.fms-inc.com](http://www.fms-inc.com)

Tom Hall [thall@fms-inc.com](mailto:thall@fms-inc.com)

Ruud Addink [raddink@fms-inc.com](mailto:raddink@fms-inc.com)