

# Validation of Method EPA 608.3 and EPA 625.1 Using Semi-Automated SPE

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## Introduction

Solid Phase Extraction (SPE) has recently gained traction to become a popular technique for sample preparation in environmental analyses. SPE works by first passing an aqueous sample through a sorbent, either in the form of a cartridge or disc, that can retain target analytes while allowing the water to pass through. A solvent is then passed through the sorbent to free the target analytes and make them available for concentration and later chromatographic analysis. Many labs are interested in SPE as a replacement for the cumbersome and solvent-heavy technique of liquid-liquid extraction (LLE). Our lab has fully transitioned from LLE to cartridge-based SPE for both our 608.3 and 625.1 analyses. The analyst Initial Demonstration of Capability (IDOC) and the Method Detection Limits (MDL) are shown in the tables.

## Instrumentation

- [Agilent 8890 GC-ECD w/ dual columns](#) for EPA 608.3
- [Agilent 7890 GC + 5975C MSD](#) for EPA 625.1
- [FMS EZ-SPE](#) for sample extraction
- [FMS Super-Vap 6 Concentrator](#) for sample concentration
- [Genevac Rocket Evaporator](#) for alternate sample concentration

## Extraction Consumables

### EPA 608.3:

- Thermo Scientific Hypersep C18 2000mg Cartridge (P/N: 60108-780)
- Supelco Supelclean Florisil/Na2SO4 Tubes (P/N: 54116-U)

### EPA 625.1:

- FMS ABN Exchange Cartridge (P/N: SPE-CAR1-ABN)
- Supelco Supelclean Coconut Charcoal SPE Tube (P/N: 57144-U)

## Results for EPA 608.3

Target Analytes	Test Conc. (µg / L)	IDOC 1 (µg / L)	IDOC 2 (µg / L)	IDOC 3 (µg / L)	IDOC 4 (µg / L)	Avg. (%) Recovery	Acceptance Range (%)	MDL (µg / L)
alpha-BHC	0.100	0.083	0.087	0.084	0.083	84%	49-130	0.003
gamma-BHC	0.100	0.085	0.088	0.085	0.081	85%	43-130	0.003
beta-BHC	0.100	0.087	0.093	0.089	0.093	91%	39-130	0.004
delta-BHC	0.100	0.086	0.091	0.085	0.095	89%	51-130	0.003
Heptachlor	0.100	0.085	0.090	0.085	0.091	88%	43-130	0.002
Aldrin	0.100	0.086	0.091	0.084	0.081	86%	54-130	0.001
Heptachlor epoxide	0.100	0.100	0.105	0.098	0.087	98%	57-132	0.004
alpha-Chlordane	0.100	0.102	0.100	0.100	0.102	101%	55-130	0.003
4,4'-DDE	0.100	0.095	0.091	0.097	0.083	92%	54-130	0.003
Endosulfan I	0.100	0.083	0.084	0.080	0.077	81%	57-141	0.003
Dieldrin	0.100	0.090	0.091	0.084	0.083	87%	58-130	0.009
Endrin	0.100	0.101	0.099	0.090	0.096	97%	51-130	0.003
4,4'-DDD	0.100	0.109	0.102	0.092	0.100	101%	48-130	0.003
Endosulfan II	0.100	0.094	0.094	0.085	0.083	89%	22-171	0.003
4,4'-DDT	0.100	0.104	0.094	0.085	0.079	91%	46-137	0.002
Endrin aldehyde	0.100	0.067	0.066	0.065	0.056	64%	23-100	0.002
Methoxychlor	0.100	0.096	0.099	0.092	0.100	97%	27-155	0.002
Endosulfan sulfate	0.100	0.090	0.092	0.086	0.081	87%	38-132	0.003
Endrin ketone	0.100	0.084	0.085	0.077	0.073	80%	47-111	0.004
Aroclor 1016	1.000	0.824	0.760	0.721	0.700	75%	61-103	0.050
Aroclor 1260	1.000	0.642	0.671	0.585	0.531	61%	37-130	0.050

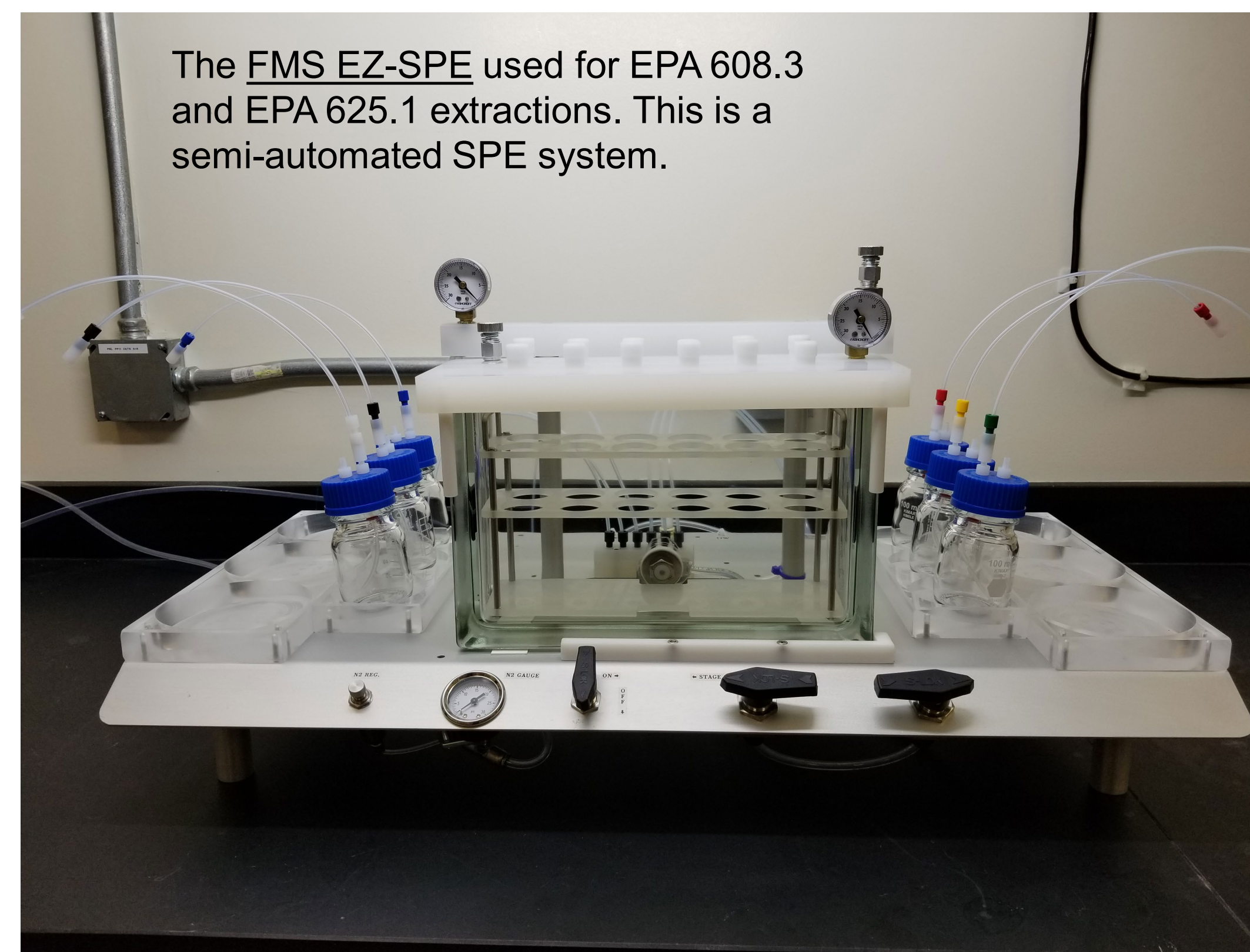
## Extraction Procedures

- **Sample Prep:** Samples are pre-weighed and tested for oxidizers and pH. Sample pH is then adjusted to less than 2.0 using 50% HCl. Surrogate and spike solution are added.
- **Extraction:** Samples are loaded on to the pre-conditioned SPE cartridges (C18 for EPA 608.3 or an ABN Exchange/charcoal cartridge stack for EPA 625.1). The sample loading is done at an approximate rate of 15mL/min. The cartridges are then dried for 10 minutes with nitrogen gas.
- **Elution:** After drying, the 608.3 cartridges are eluted with 50mL of a 10% acetone/90% hexane mix passing through Florisil/Sodium Sulfate clean-up cartridges into collection vials. For 625.1, the cartridges are each eluted with 30-40mL of methylene chloride into a collection vial.
- **Base Adjustment (EPA 625.1):** The ABN Exchange cartridges are next treated with 30mL of 1% NaOH. After this base treatment, the ABN cartridges are eluted a second time with 30-40mL of methylene chloride to collect the base fraction.

## Results for EPA 625.1\*

Target Analytes	Test Conc. (µg / L)	IDOC 1 (µg / L)	IDOC 2 (µg / L)	IDOC 3 (µg / L)	IDOC 4 (µg / L)	Avg. (%) Recovery	Acceptance Range (%)	MDL (µg / L)
N-Nitrosodimethylamine	20.00	11.77	13.86	13.59	14.66	67%	2-150	1.08
Phenol	20.00	13.26	15.83	14.90	15.86	75%	17-120	0.16
bis(2-chloroisopropyl)ether	20.00	15.89	19.04	18.23	19.12	90%	63-139	0.16
n-Nitroso-di-n-propylamine	20.00	17.40	20.57	19.60	19.41	96%	14-198	0.24
p-Cresol	20.00	14.04	16.67	15.82	16.18	78%	10-125	0.15
Hexachloroethane	20.00	11.65	13.99	13.55	14.49	67%	55-120	0.13
bis(2-Chloroethoxy)methane	20.00	13.84	16.51	15.89	15.90	78%	49-165	0.11
Naphthalene	20.00	13.54	16.04	15.45	15.55	76%	36-120	0.12
Hexachlorocyclopentadiene	20.00	7.40	9.87	10.95	9.48	47%	2-125	0.68
2,4,5-Trichlorophenol	20.00	13.71	16.50	15.60	15.06	76%	37-144	0.27
2-Chloronaphthalene	20.00	13.47	16.23	15.96	14.93	76%	65-120	0.18
2,4-Dinitrophenol	20.00	12.43	13.82	13.56	14.24	68%	D-173	1.78
4-Nitrophenol	20.00	12.08	13.39	13.60	14.10	66%	13-129	0.77
Diethylphthalate	20.00	16.78	17.74	18.43	18.10	89%	D-120	0.16
Fluorene	20.00	14.46	16.59	16.36	15.48	79%	70-120	0.16
4,6-Dinitro-2-methylphenol	20.00	16.02	17.43	17.64	18.16	87%	53-130	0.39
Azobenzene	20.00	14.06	16.13	15.75	15.40	77%	10-150	0.22
Pentachlorophenol	20.00	14.79	15.78	16.12	16.89	79%	38-152	0.28
Benzidine	20.00	5.88	10.11	9.35	11.68	46%	2-150	0.68
3,3'-Dichlorobenzidine	20.00	6.23	6.35	6.42	7.20	33%	8-213	0.61
Chrysene	20.00	17.29	18.36	19.06	18.73	92%	44-140	0.27
Benzo(b)fluoranthene	20.00	18.21	20.40	21.14	20.07	100%	42-140	0.24
Indeno(1,2,3-cd)pyrene	20.00	16.38	17.53	18.45	18.72	89%	D-151	0.28
Benzo(g,h,i)perylene	20.00	16.39	17.57	18.43	18.64	89%	D-195	0.29

\* This is just a partial list of compounds analyzed, please reach out to presenter for more info.



The [FMS EZ-SPE](#) used for EPA 608.3 and EPA 625.1 extractions. This is a semi-automated SPE system.



## Conclusions

The City of San Jose (CSJ) laboratory was able to fully validate EPA methods 608.3 and 625.1 using a cartridge-based SPE. Following SPE method validation, we have analyzed industrial compliance samples, plant effluent/influent, and Performance Testing (PT) samples with great success. By extracting with SPE instead of LLE we have greatly reduced both time and solvent used by our extractions.

## References

EPA Method 608.3 Organochlorine Pesticides and PCBs by GC/HSD, United States Environmental Protection Agency, December 2016, [www.epa.gov/sites/default/files/2017-08/documents/method\\_608-3\\_2016.pdf](http://www.epa.gov/sites/default/files/2017-08/documents/method_608-3_2016.pdf)

EPA Method 625.1 Base/Neutrals and Acids by GC/MS, United States Environmental Protection Agency, December 2016, [https://www.epa.gov/sites/default/files/2017-08/documents/method\\_625-1\\_2016.pdf](https://www.epa.gov/sites/default/files/2017-08/documents/method_625-1_2016.pdf)

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