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A Comparison of the Use of Microwave Extraction for Organics – SW-846, Method 3546

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NEMC, Orange County, CA August 8-12, 2016

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What is method 3546?

- Microwave Extraction is a relatively new method.
- The sample and solvent are placed in a Teflon™ extraction vessel. The vessel is sealed and subjected to microwave energy for a set amount of time.
- Microwave energy raises the temperature of the solvent in the closed vessel. The closed vessel allows pressure to rise and the solvent remains a liquid even above its boiling point.
- This allows the solvent to be efficient at extracting the desired analytes from the solid sample.

What is method 3546?

- The equipment allows for extraction of up to 40 samples at a time. This allows for an increased preparation capacity and a decreased sample turn-around time.

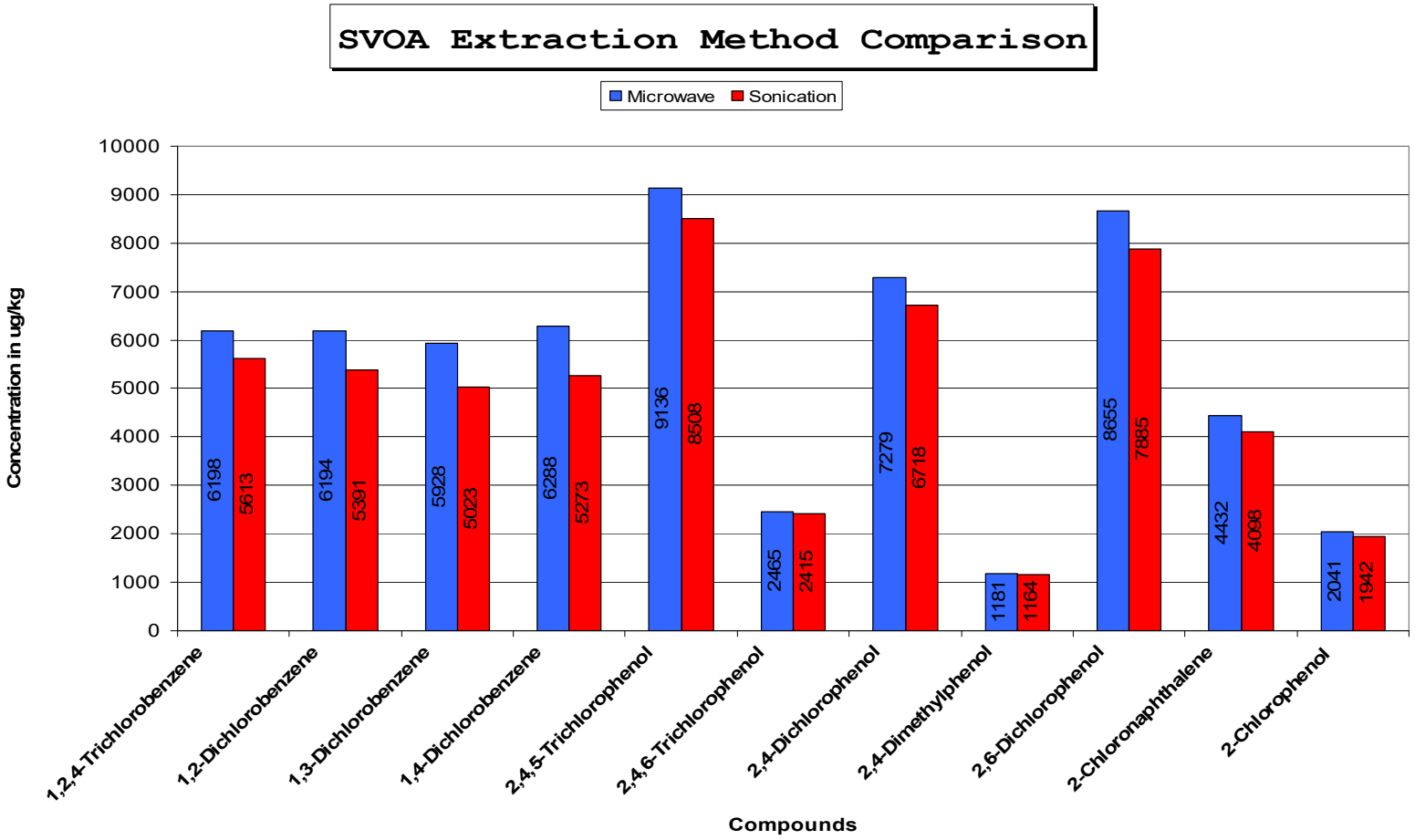
What is the alternative?

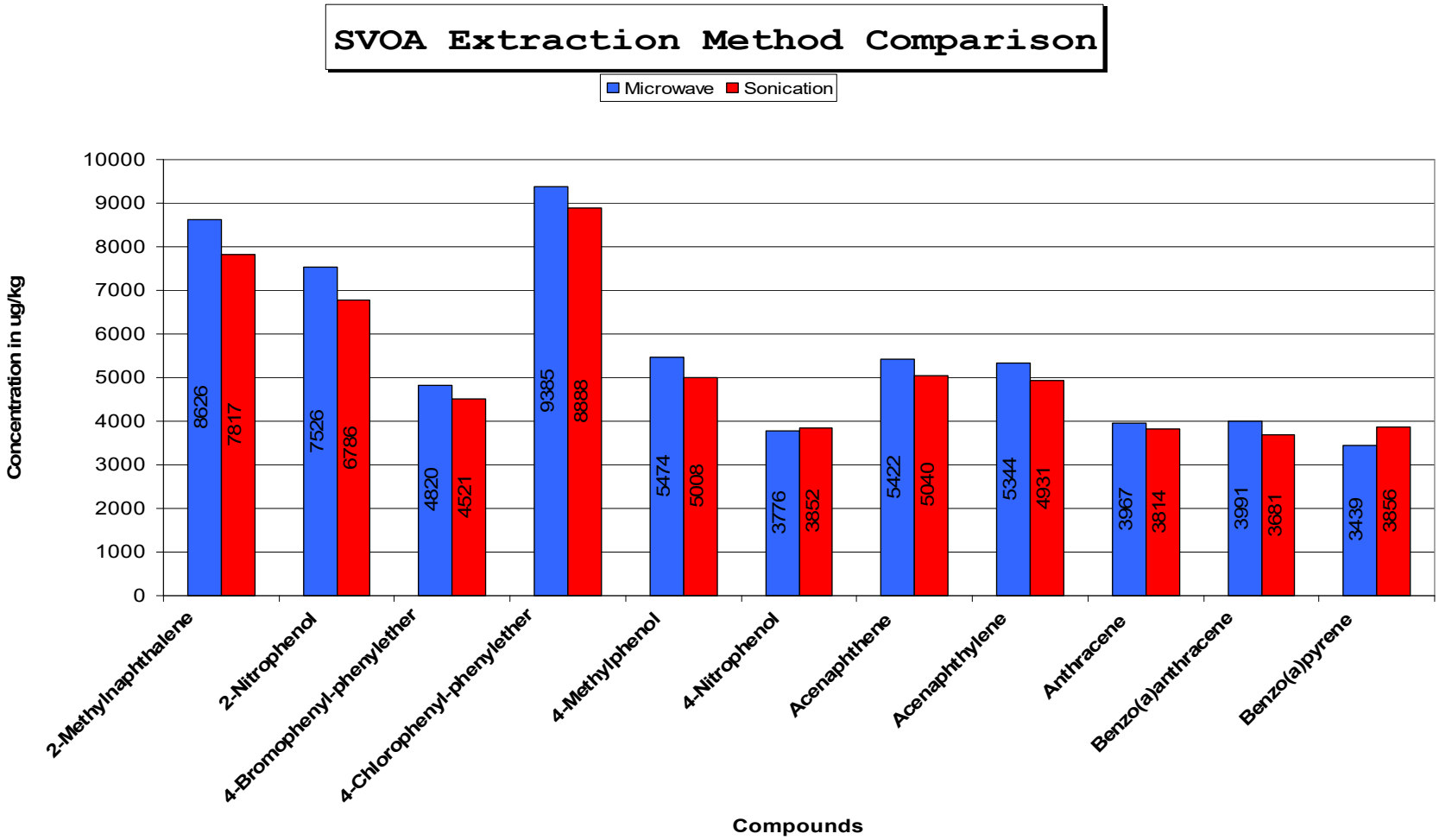
- Ultrasonic Extraction EPA Method 3550
- The solid sample is mixed with sodium sulfate in a beaker and solvent is added.
- Ultrasonic disrupters are used to facilitate contact between the solid sample and solvent.
- This extraction procedure allows for the extraction of 3 samples at a time.

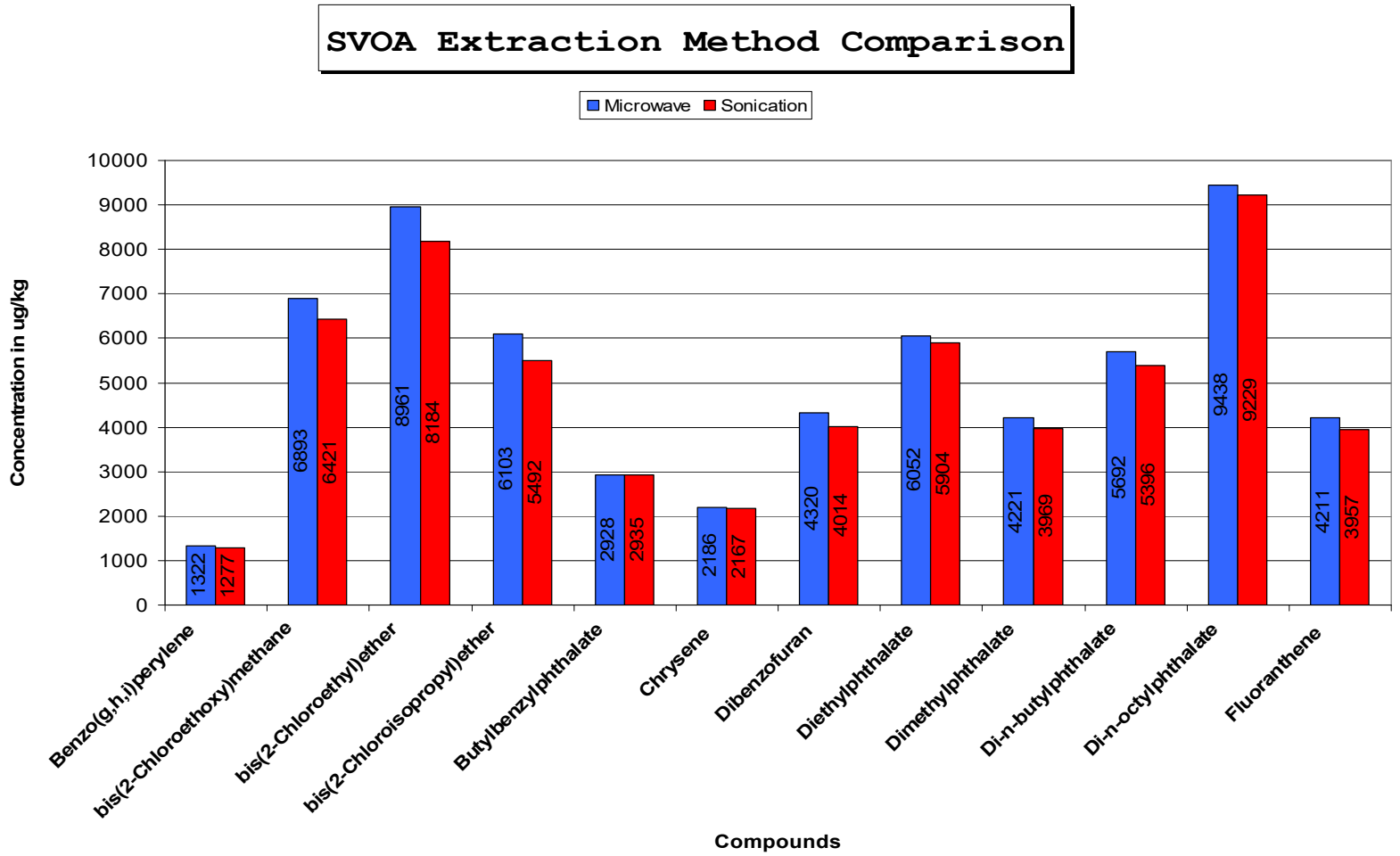
How do these two methods compare?

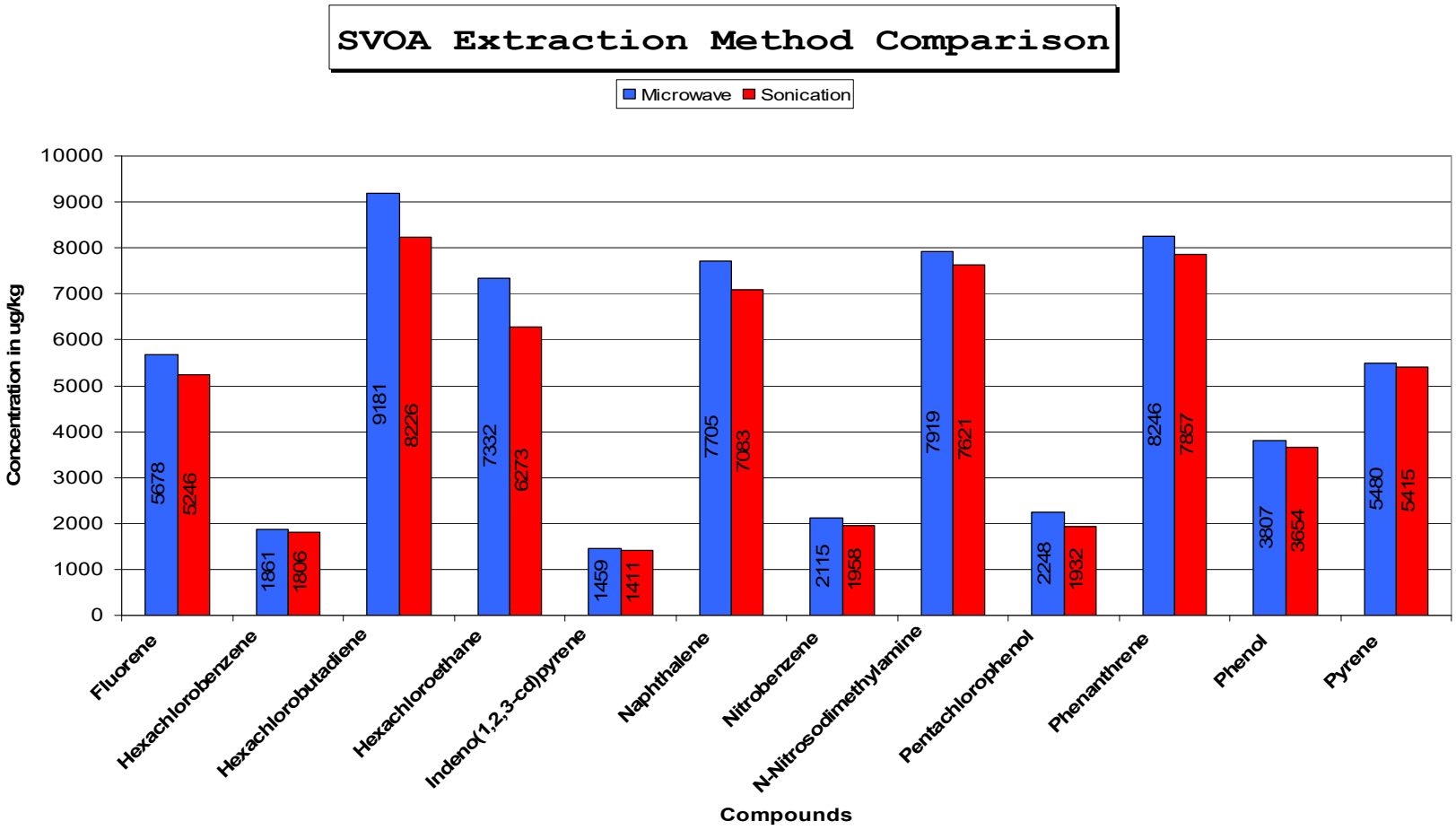
- Microwave Extraction uses **ten times** less solvent than Ultrasonic Extraction. Microwave Extraction allows for a “greener” approach to the preparation of environmental samples.
- The sample handling and concentration times are greatly reduced when 3546 is used.
- The preparation capacity of 3546 is 2 to 3 times greater than 3550.
- The data produced from these two preparation techniques is very comparable.


MARS
39
40
17
18
19
20
21


SVOA Extraction Method Comparison
Microwave
Sonication
Concentration in ug/kg
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
6198
5613
6194
5391
5928
5023
6288
5273
9136
8508
2465
2415
7279
6718
1181
1164
8655
7885
4432
4098
2041
1942
1,2,4-Trichlorobenzene
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
2,4,5-Trichlorophenol
2,4,6-Trichlorophenol
2,4-Dichlorophenol
2,4-Dimethylphenol
2,6-Dichlorophenol
2-Chloronaphthalene
2-Chlorophenol
Compounds


SVOA Extraction Method Comparison
Microwave
Sonication
Concentration in ug/kg
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
8626
7817
7526
6786
4820
4521
9385
8888
5474
5008
3776
3852
5422
5040
5344
4931
3967
3814
3991
3681
3439
3856
2-Methylnaphthalene
2-Nitrophenol
4-Bromophenyl-phenylether
4-Chlorophenyl-phenylether
4-Methylphenol
4-Nitrophenol
Acenaphthene
Acenaphthylene
Anthracene
Benzo(a)anthracene
Benzo(a)pyrene
Compounds


SVOA Extraction Method Comparison
Microwave
Sonication
Concentration in ug/kg
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
1322
1277
6893
6421
8961
8184
6103
5492
2928
2935
2186
2167
4320
4014
6052
5904
4221
3969
5692
5396
9438
9229
4211
3957
Benzo(g,h,i)perylene
bis(2-Chloroethoxy)methane
bis(2-Chloroethyl)ether
bis(2-Chloroisopropyl)ether
Butylbenzylphthalate
Chrysene
Dibenzofuran
Diethylphthalate
Dimethylphthalate
Di-n-butylphthalate
Di-n-octylphthalate
Fluoranthene
Compounds


SVOA Extraction Method Comparison
Microwave
Sonication
Concentration in ug/kg
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
5678
5246
1861
1806
9181
8226
7332
6273
1459
1411
7705
7083
2115
1958
7919
7621
2248
1932
8246
7857
3807
3654
5480
5415
Fluorene
Hexachlorobenzene
Hexachlorobutadiene
Hexachloroethane
Indeno(1,2,3-cd)pyrene
Naphthalene
Nitroberzene
N-Nitrosodimethylamine
Pentachlorophenol
Phenanthrene
Phenol
Pyrene
Compounds

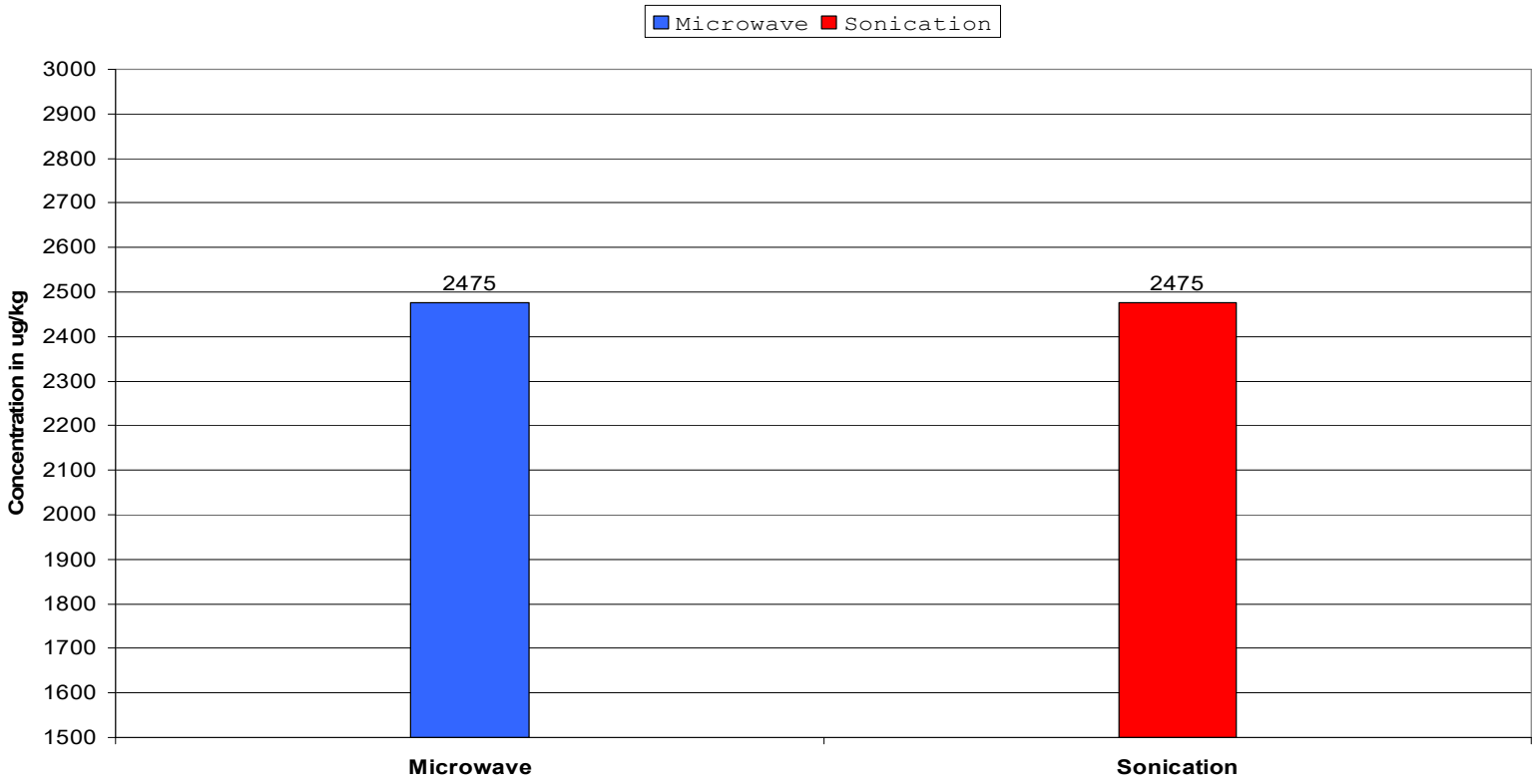
Performance Limits

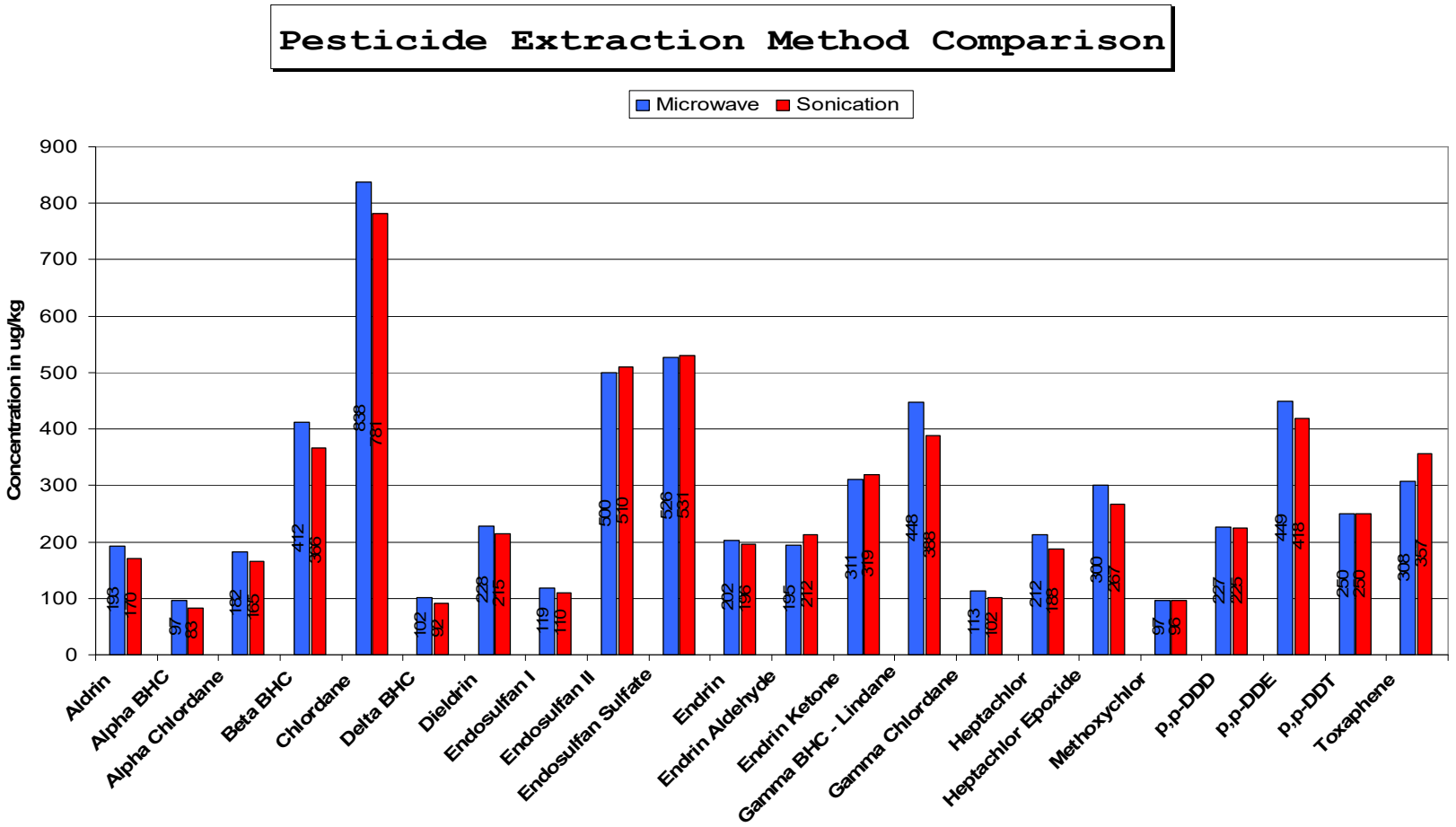
Compound	Microwave limits	DoD Limits
Phenol	73-122	34-121
2-Chlorophenol	85-123	34-121
N-Nitroso-di-n-propylamine	67-121	36-120
Nitrobenzene	70-122	34-122
2-Nitrophenol	83-120	36-123
Naphthalene	82-112	35-123
4-Chloroaniline	10-100	17-106
Dimethylphthalate	82-113	48-124
3-Nitroaniline	66-119	33-119
2,4-Dinitrophenol	16-132	27-140
2,4-Dinitrotoluene	81-122	48-126
Hexachlorobenzene	79-116	45-122
N-Nitrosodiphenylamine	83-118	38-127
Pentachlorophenol	57-126	25-133

Performance Limits

Compound	Microwave limits	DoD Limits
4-Bromophenyl-phenylether	84-120	46-124
Phenanthrene	80-114	50-121
Di-n-butylphthalate	84-120	51-128
Fluoranthene	81-117	50-127
Butylbenzylphthalate	80-118	48-132
3,3'-Dichlorobenzidene	10-116	22-121
Benzo(a)anthracene	76-119	49-126
Bis(2-Ethylhexyl)phthalate	81-121	51-133
Di-n-octylphthalate	80-140	45-140
Benzo(a)pyrene	85-117	45-129
Benzo(g,h,i)perylene	77-118	43-134

PCB Extraction Method Comparison
Aroclor - 1254




Pesticide Extraction Method Comparison
Microwave
Sonication
Concentration in ug/kg
0
100
200
300
400
500
600
700
800
900
193
170
97
83
182
165
412
366
838
781
102
92
228
215
119
110
500
510
526
531
202
196
195
212
311
319
448
388
113
102
212
188
300
267
97
95
227
225
449
418
250
250
308
357
Aldrin
Alpha BHC
Alpha Chlordane
Beta BHC
Chlordane
Delta BHC
Dieldrin
Endosulfan I
Endosulfan II
Endosulfan Sulfate
Endrin
Endrin Aldehyde
Endrin Ketone
Gamma BHC - Lindane
Gamma Chlordane
Heptachlor
Heptachlor Epoxide
Methoxychlor
p,p-DDD
p,p-DDE
p,p-DDT
Toxaphene

Performance Limits

Compound	Microwave limits	DoD Limits
Endrin Aldehyde	59 - 122	35 - 137
Endrin Ketone	64 - 121	55 - 136
Alpha Chlordane	73 - 131	54 - 133
Gamma Chlordane	76 - 134	53 - 135
Alpha BHC	65 - 124	45 - 137
Beta BHC	68 - 129	50 - 136
Gamma BHC - Lindane	47 - 140	49 - 135
Delta BHC	45 - 151	47 - 139
Heptachlor	66 - 118	47 - 136
Aldrin	60 - 117	45 - 136
Heptachlor Epoxide	74 - 128	52 - 136
p,p-DDE	68 - 146	56 - 134
p,p-DDD	69 - 138	56 - 139
p,p-DDT	67 - 135	50 - 141

Performance Limits

Compound	Microwave limits	DoD Limits
HCB	80 - 120	57 - 126
Mirex	75 - 125	65 - 128
Methoxychlor	65 - 131	52 - 143
Dieldrin	63 - 126	56 - 136
Endrin	65 - 125	57 - 140
Chlordane	75 - 125	43 - 149
Toxaphene	70 - 120	33 - 141
Endosulfan I	62 - 119	53 - 132
Endosulfan II	65 - 126	53 - 134
Endosulfan Sulfate	71 - 132	55 - 136
PCB-1016	76 - 121	47 - 134
PCB-1254	60 - 130	67 - 135
PCB-1260	79 - 130	53 - 140

Questions