



Air Monitoring Applications Using XAD-2[®] And Silica Gel Adsorbents In a Timesaving Air Sampling Solid Phase Extraction Tube (ASSET[™])

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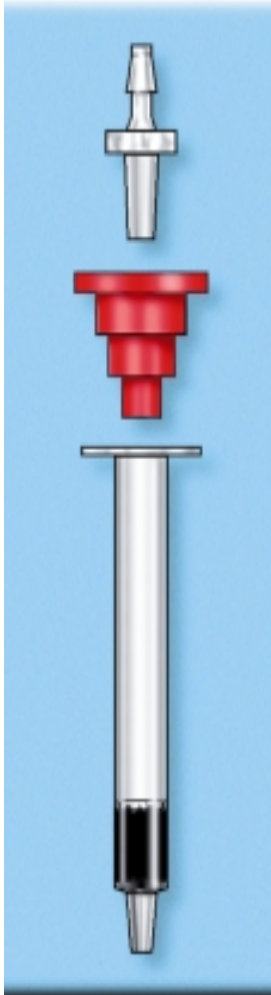
Abstract

The ASSET (Air Sampling Solid Extraction Tube) is an air sampling device in the form of a solid phase extraction (SPE) tube in which extraction is performed by gravity-feed elution, rather than removal of the adsorbent followed by solvent desorption, that is typical for glass tubes. This timesaving feature has been utilized in the development of two new applications: one for PAHs using XAD-2 (ASSET-43) as the adsorbent, and one for nitrobenzene using silica gel (ASSET-52).

Following NIOSH 5515 the ASSET-43 was evaluated for naphthalene and benzo(a)pyrene by GC/FID and comparisons made with a standard glass tube containing the same amount of XAD-2. NIOSH 2005 was evaluated for nitrobenzene with the same comparisons made with a glass tube. Background levels, a recovery study over 4 concentration levels, and a storage stability study was performed for both tubes. Performance criteria were targeted at $\leq 10\%$ difference to the glass tube equivalent. Results of the validation indicate that the performance of the ASSET is comparable to the corresponding glass sampling tube for both applications in all parameters investigated.

In addition, a round robin was conducted with several AIHA accredited laboratories to assist in the validation of the ASSET-43 and ASSET-52. The overall mean recovery of nitrobenzene in a set of 6 spiked tubes was 92%. The results from the PAH round robin yielded low recoveries, particularly for benzo(a)pyrene, for labs using HPLC rather than GC for the analysis.

The ASSET offers many advantages, including the convenience of a polypropylene tube and timesaving sample prep, while demonstrating equivalent performance to the traditional glass sorbent tube.



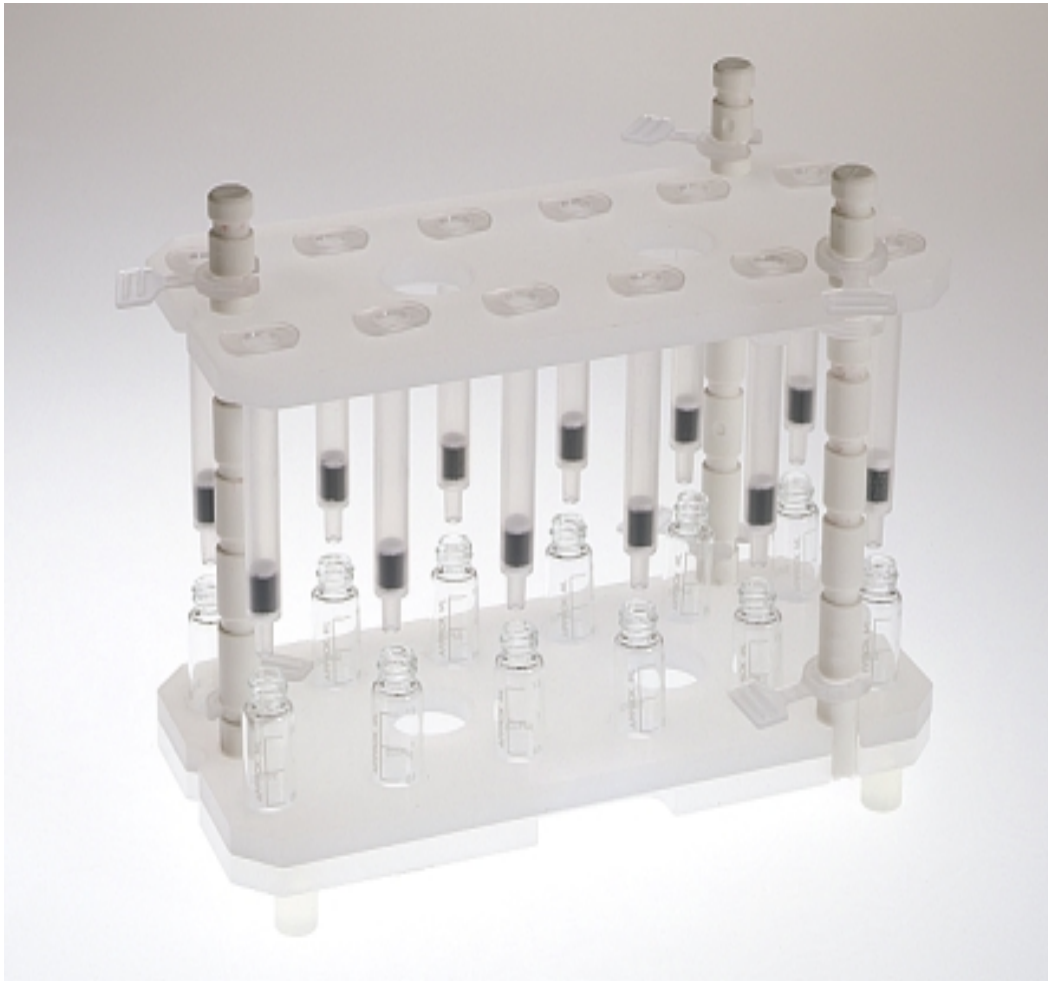
Air Sampling Solid Extraction Tube



**Sample
Collection
Using Dual
ASSET
Tubes**

**With and
Without Backup**

Sample Desorption by Gravity Elution



- **Elute 12 samples simultaneously**
- **Elute directly into GC vials**

Tube Specifications

	ASSET-43	ASSET-52
Adsorbent	20/40 mesh XAD-2	45/60 mesh silica gel
Bed Weight	100 mg	150 mg
Method Application	NIOSH 5515	NIOSH 2005
Compounds Tested	Naphthalene Benzo(a)pyrene	Nitrobenzene

Experimental Procedures

- **Spiking Technique**
 - **direct liquid spikes of dilute mixtures in eluent**
 - **concentrations of 20, 200, 1000, 2000 μ g/tube**
 - **spiked tubes left at room temperature overnight before analysis**
- **Sample Prep**
 - **ASSET-43 gravity elution with 5mL toluene**
 - **ASSET-52 gravity elution with 1mL methanol with 10 min solvent residence time**

ASSET-43 PAHs by NIOSH 5515

ASSET-43 spiked with 200ug of naphthalene & benzo(a)pyrene

Analytical Conditions

SPB-5 capillary column

30m x 0.53mm ID x
1.5 μ m film

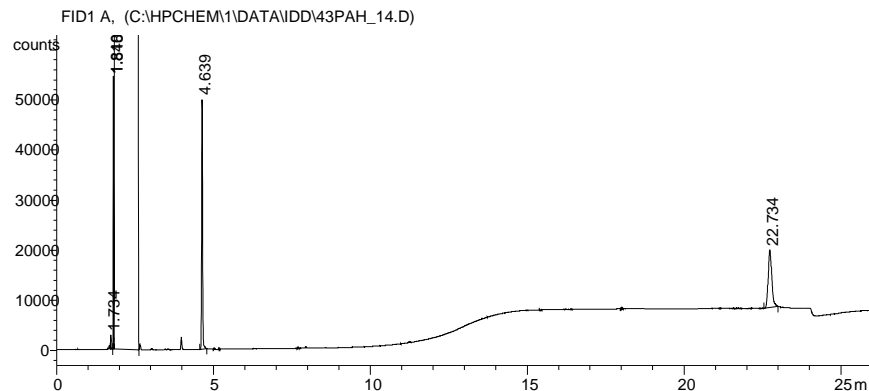
Helium, 8ml/min

150°C for 3 min, 15°C/min to
300°C, hold 13 min

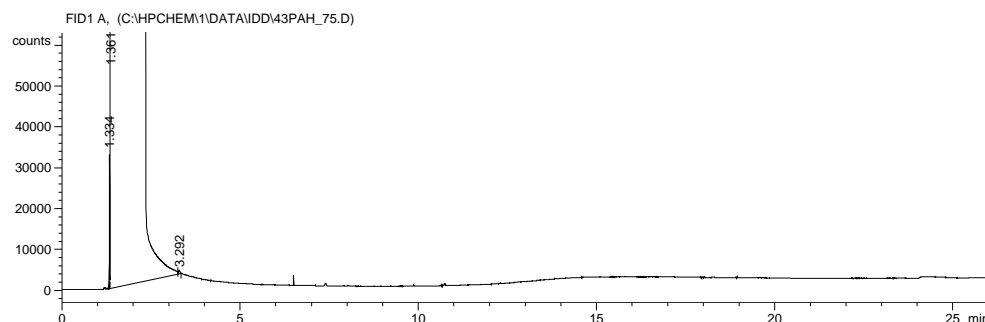
FID @ 320°C

Splitless, direct inj @ 220°C

1 μ L injection



Blank ASSET-43 desorbed in toluene



ASSET-52 EXPLOSIVES by NIOSH 2005

Analytical Conditions

SUPELCOWAX-10 capillary column

30m x 0.53mm ID x 0.5 μ m film

Helium, 8ml/min

90°C for 0 min, 8°C/min to 170°C,

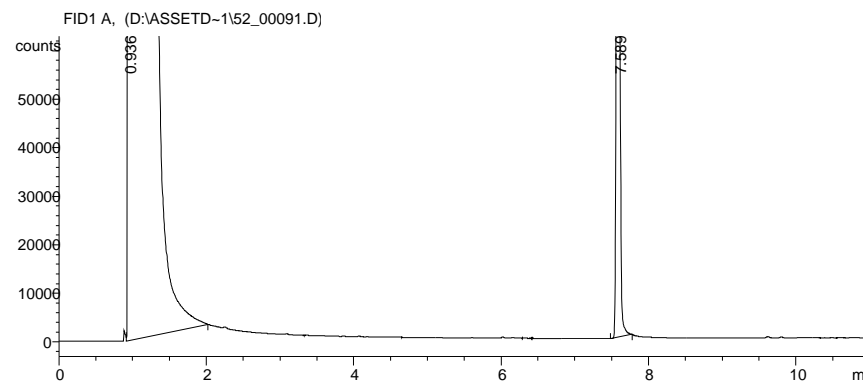
hold 1 min

FID @ 300°C

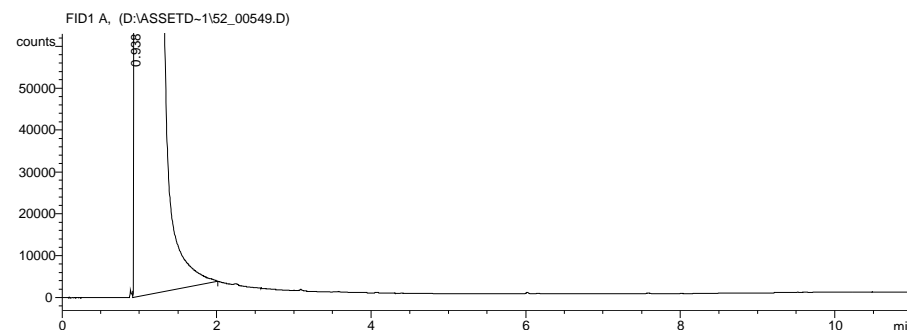
Packed, direct inj @ 260°C

1 μ L injection

ASSET-52 spiked with 200ug of nitrobenzene

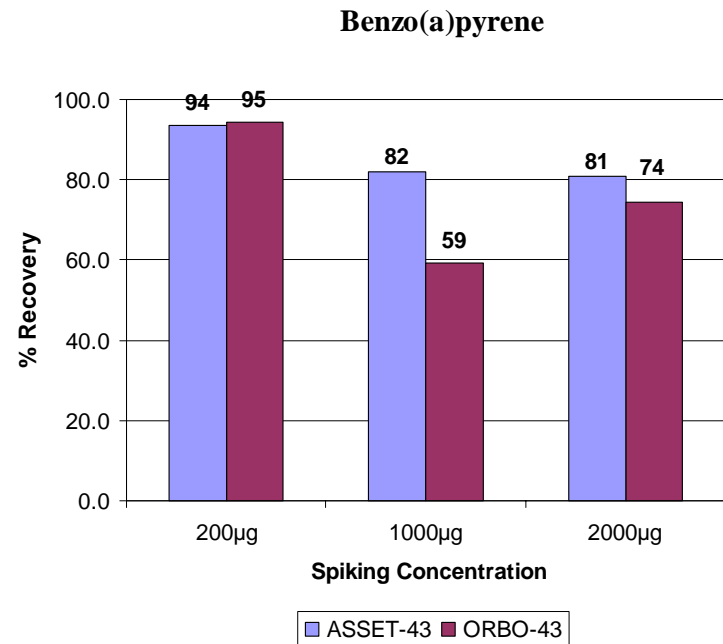
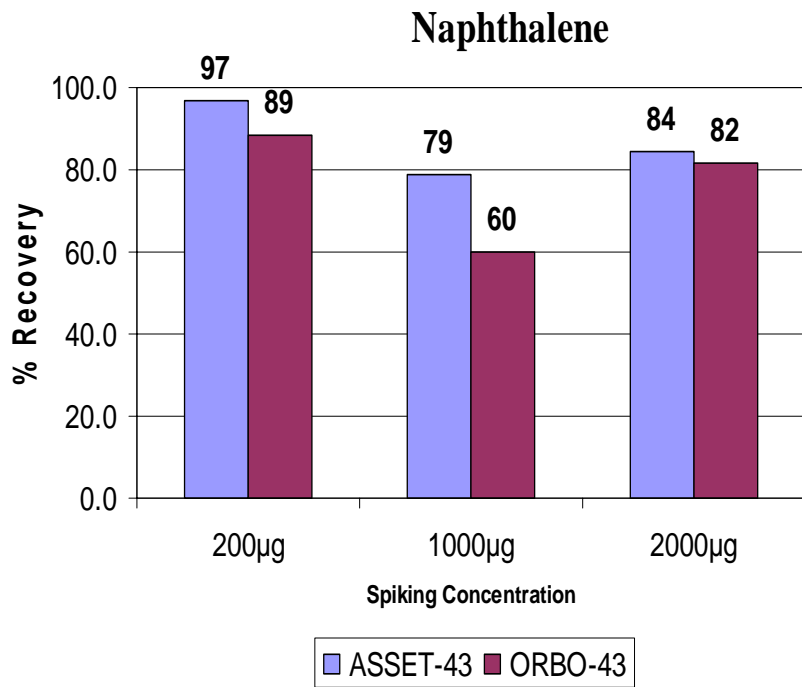


Blank ASSET-52 desorbed in methanol

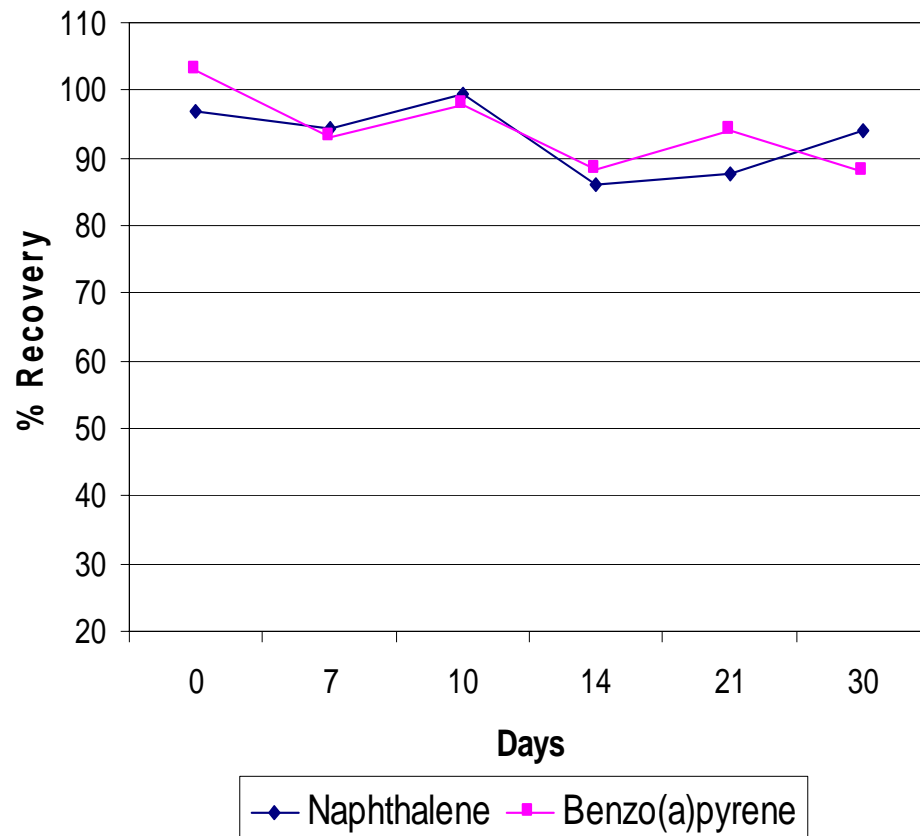


ASSET-43 Recovery

Comparison of ASSET & ORBO



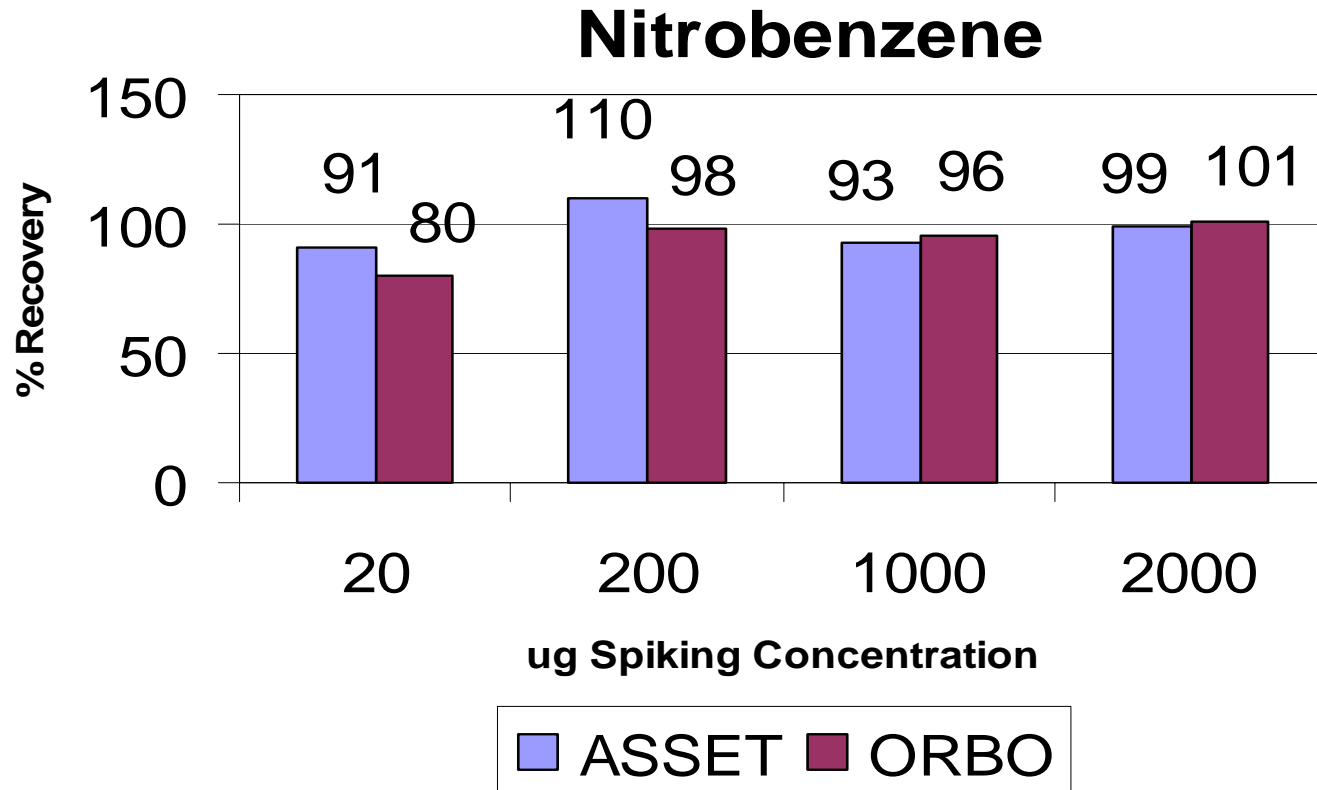
ASSET-43 Stability Study



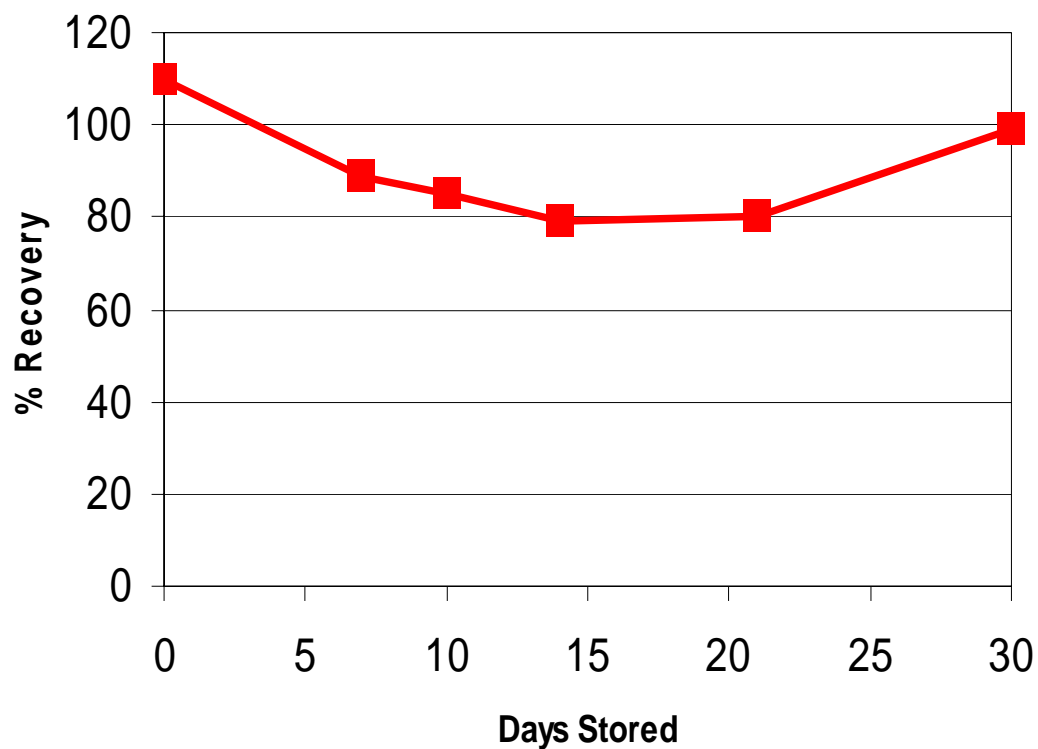
Tubes were spiked with 200ug of each compound and stored for up to 30 days under refrigeration.

ASSET-52 Recovery

Comparison of ASSET & ORBO



ASSET-52 Stability Study



Tubes were spiked with 200ug of nitrobenzene and stored for up to 30 days under refrigeration.

ASSET Round Robin

- **Several accredited labs participated in an interlaboratory study consisting of a set of 6 spiked tubes and 1 blank**
- **Participating Laboratories**
 - **Clayton**
 - **DataChem**
 - **Galson**
 - **Kemper-NATLSCO**
 - **LabCorp Analytics**
 - **NIOSH**
 - **Wisc Occupational Health Lab**

ASSET-43 Round Robin Results

Overall Mean Recovery = 79%

Overall Std Dev = 16

Overall RDS = 20%

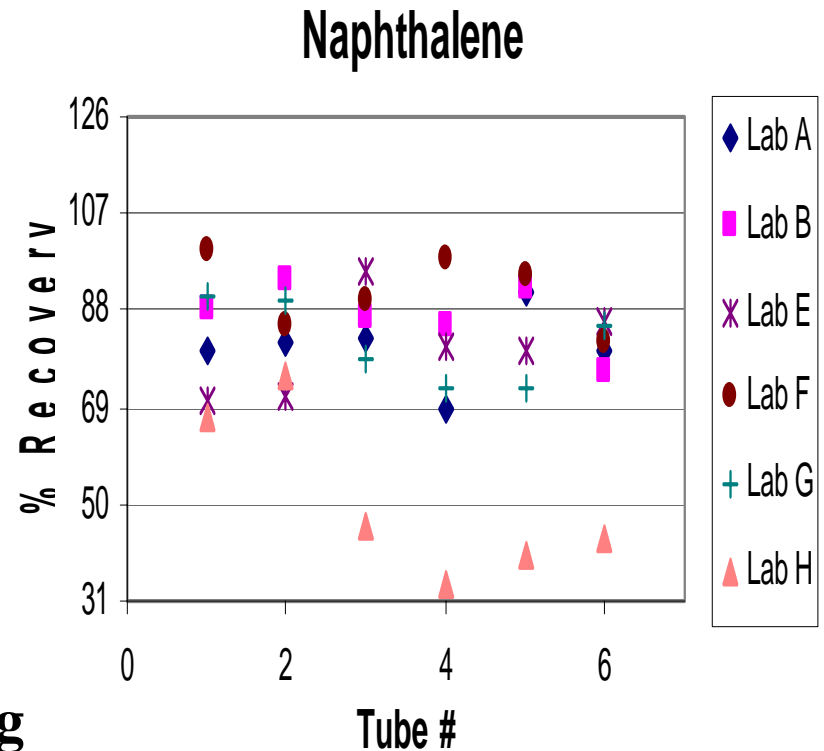
UCL (3σ) = 127

LCL (3σ) = 31

Outliers = 0

Duplicate Tubes = 1&3, 2&5, 4&6

Spike Conc respectively = 40, 200, 1000ug



ASSET-43 Round Robin Results

Overall Mean Recovery = 38%

Overall Std Dev = 14

Overall RDS = 37%

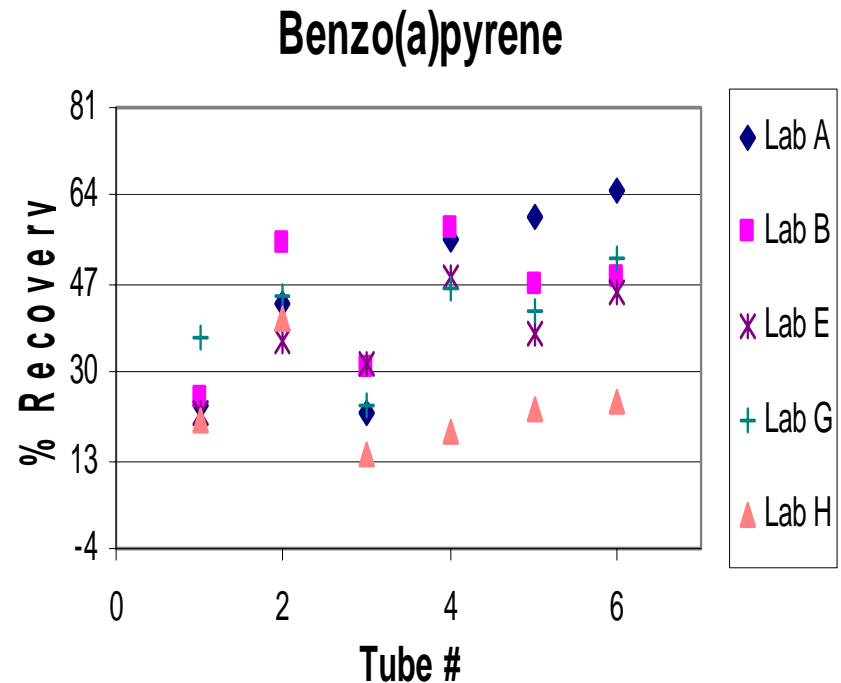
UCL (3σ) = 80

LCL (3σ) = -4

Outliers = 0

Duplicate Tubes = 1&3, 2&5, 4&6

Spike Conc respectively = 40, 200, 1000ug



ASSET-52 Round Robin Results

Overall Mean Recovery = 92%

Overall Std Dev = 31

Overall RDS = 34%

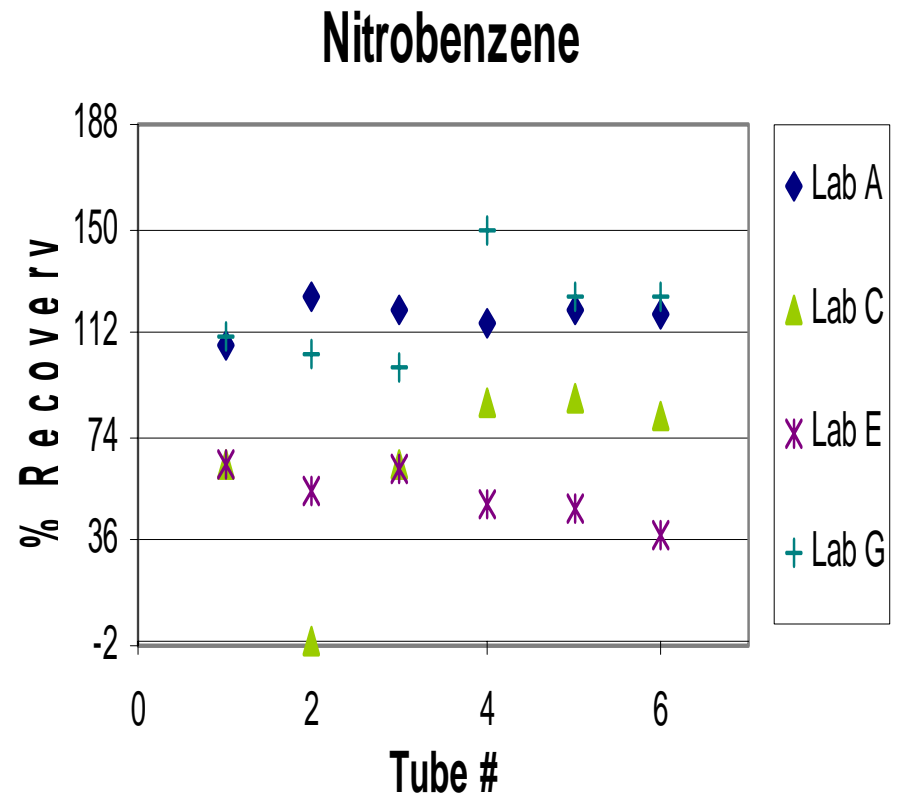
UCL (3 σ) = 186

LCL (3 σ) = -2

Outliers = 0

Duplicate Tubes = 4&5, 2&6, 1&3

Spike Conc respectively = 40, 200, 1000ug



CONCLUSION

- **ASSET tubes demonstrate equivalent performance to glass ORBO tubes**
- **Significant timesaving in sample prep is realized with ASSET elution versus glass tube desorption**
- **NIOSH methods can be applied to ASSET tubes by only changing the desorption technique**
- **With no glass, ASSET tubes are safe and convenient to handle both in the field and in the lab**
- **Single-bed ASSET tubes only require one analysis**
- **ASSET tubes are automatable with robotics sample prep station**