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Overview of the Technology and Best Practices for Sample Collection and Preservation

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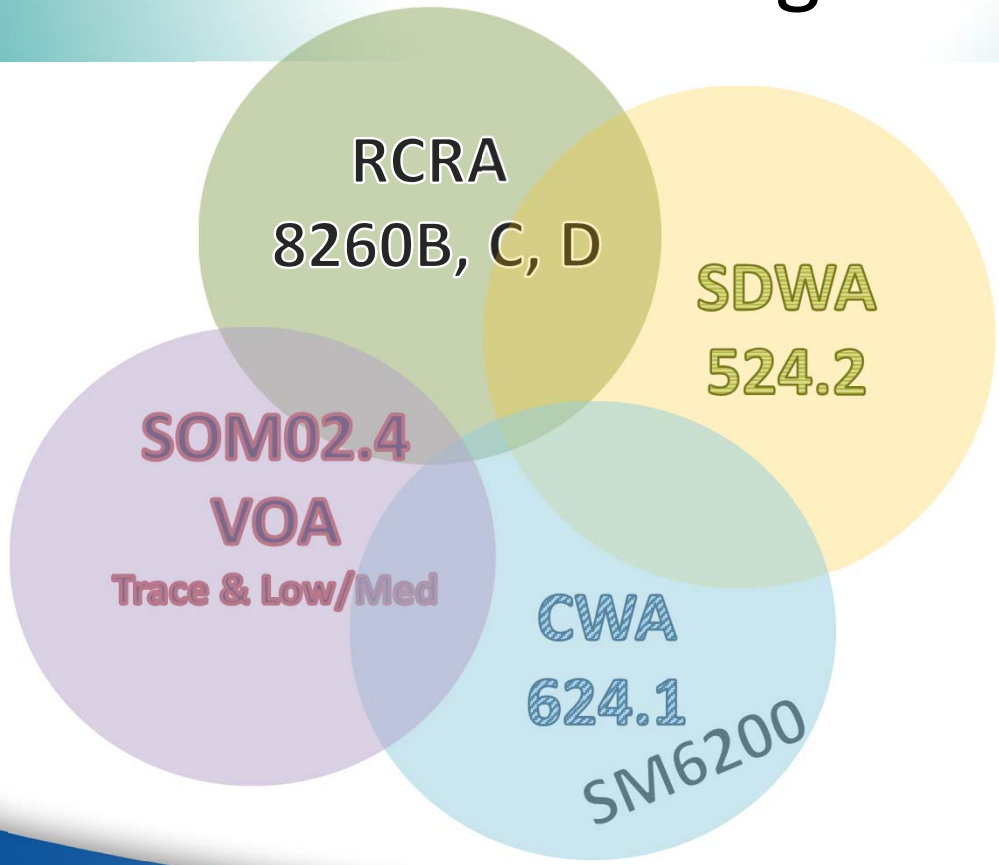
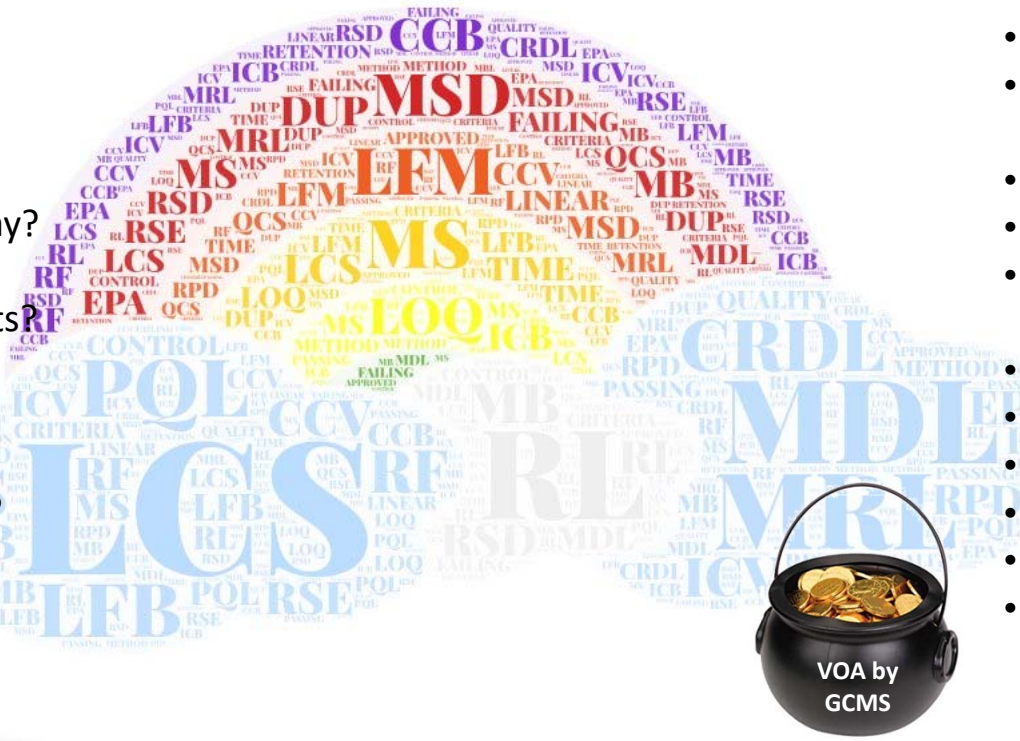
Six Methods…..Four Programs



Just Imagine…..if there were only ONE Method

QUESTIONS

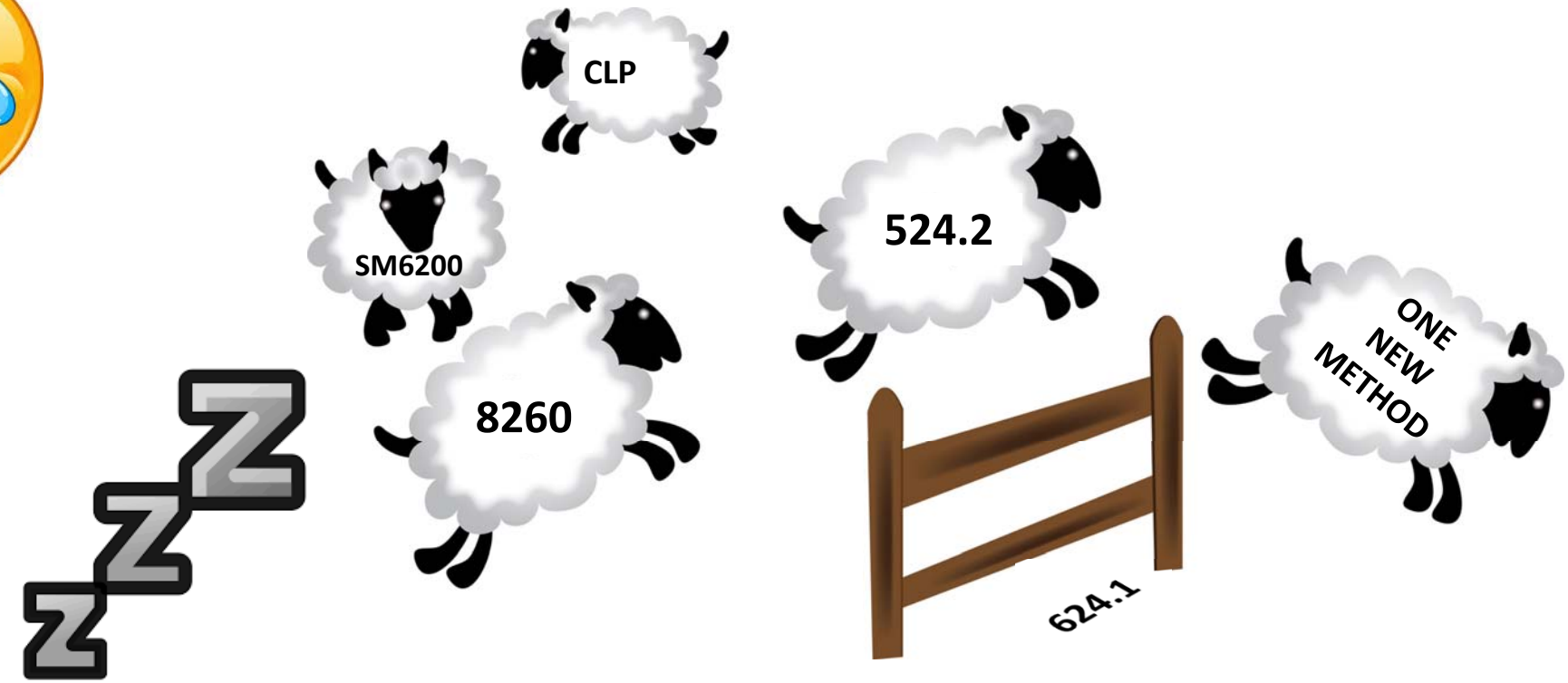
- Weighted? Not Weighted?
- 2nd Source? Primary Source?
- LFB? LCS?
- LOQ? CRDL? RL? MRL?
- RSD? 15%, 20%, 30%
- RF? Linear? Options???
- Tune? There's more than 1 way?
- MDL? LOD?
- In-house limits? Method limits?
- Suggested? Recommended? Required?
- Qualify? Okay? Not Allowed?
- Recalc curve pts back to ICAL?
- % D, Recovery? Etc.
- Verify ICV, CCV, LCS, MS/MSD
- 12 hour? 24 Hour?



ANSWERS

- Standardized terms
- One set of criteria
- Performance Appropriate
- Holding time..3,7,14
- Cal Points - 5
- Cal Conc – Range established for use
- Internal Stds
- Surrogates (not DMCs)
- Batch Criteria – set
- Time (12 or 24 hr) - set
- QC Frequency – set
- Sensitivity Ck - Set

WAKE UP! …..You're Dreaming



Finding the Foundation

Goal of VOA Analysis and GCMS Technology

Common PURPOSE

- To identify purgeable organic pollutants in all types of water, including but not limited to the following: ground water, surface water, industrial (permitted) discharge, waste water, storm water, drinking water, estuaries, rivers, streams, lakes, etc.
- Many contamination issues are traceable to any of the following: leaking underground fuel or solvent storage vessels, landfills, agriculture practices, and wastewater disposal.
- The most probable cause for the contamination of some aquifers and surface waters has never been firmly established.

Finding the Foundation

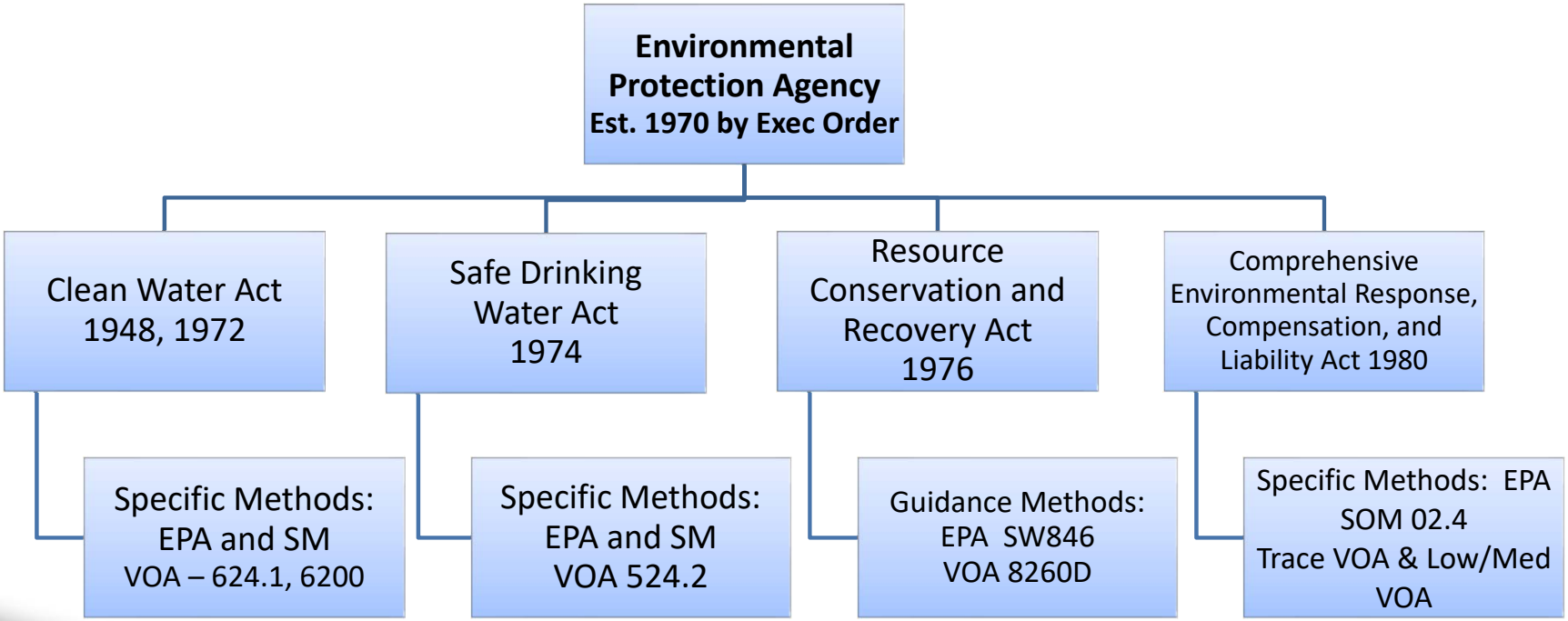
Goal of VOA Analysis and GCMS Technology

Summary of Methods

- Inert gas is bubbled through a known volume of water via a purging chamber.
- Target analytes are efficiently transferred from aqueous to vapor phase
- Vapor enters a sorbent trap where the purgeables are trapped
- The trap is heated & backflushed with the gas to desorb the purgeables onto a gas chromatographic column.
- The column is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.

Common technique...

Water Related Programs & Methods



Sample Collection - Field

Method	Blank Required	Container	#
524.2	(2) Field Reagent Blanks	40mL vial* w/TFE septum	2
624.1	NA	25–40mL vial* w/TFE septum	NS
6200	(2) Field Reagent Blanks	25–40mL vial* w/TFE septum	2
8260 D	Trip Blank Recommended	40mL vial* w/TFE septum	2
CLP Trace	Storage Blank Required	40mL vial	3
CLP Low Med	Storage Blank Required	40mL vial	3

* Wash vials, caps, and septa with detergent, rinse, and dry at 105°C for 1 h, before use in an area free of organic vapors.

Sample Preservation

Method	Unpres	Pres <2 HCl	Chlorinated
524.2	24 hour	14 d	Dechlorinate * prior to adding acid
624.1	7 d (Acrolein – 3 d)	14 d	"
6200	NA	14 d	"
8260C, D	7 d (2-CEVE ASAP)	14 d	"
CLP Trace	NA	10 d from rcpt.	NA
CLP Low/med	24 hr	10 d from rcpt.	NA

* Ascorbic Acid, Sodium thiosulfate

Blank Criteria

Method	Detection Levels	Purge Volume
524.2	<MDL	25mL
624.1	<MDL, 1/3 Reg Limit, <1/10 conc of spl	5-25mL
6200	<MQL	25mL
8260 D	<1/2 LLOQ or <10% of conc of spls	5mL
CLP Trace	<CRQL	25mL
CLP Low Med	<CRQL	5mL

Sample Processing

Method	Batch Processing	Calibration Levels
524.2	QC is per 12 hour Shift	3
624.1	QC is per 12 hour Shift	5
6200	QC is per 12 hour Shift	5
8260C, D	QC is per 12 hour Shift	5
CLP Trace	QC is per 12 hour Shift	5
CLP Low Med	QC is per 12 hour Shift	5

Total Analytes Represented =139

Method	Analytes
524.2	84
624.1	93
6200	61
8260D	128
CLP Trace	51
CLP Low Med	51

Analytes

38 Analytes appear in ALL Methods

1,1,1-Trichloroethane	71-55-6
1,1,2,2-Tetrachloroethane	79-34-5
1,1,2-Trichloroethane	79-00-5
1,1-Dichloroethane	75-34-3
1,1-Dichloroethene (Vinylidene chloride)	75-35-4
1,2,3-Trichlorobenzene	87-61-6
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8
1,2-Dibromoethane (EDB, Ethylene dibromide)	106-93-4
1,2-Dichlorobenzene	95-50-1
1,2-Dichloroethane	107-06-2
1,2-Dichloropropane	78-87-5
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
Benzene	71-43-2
Bromochloromethane	74-97-5
Bromodichloromethane	75-27-4
Bromoform	75-25-2
Bromomethane	74-83-9
Carbon tetrachloride	56-23-5

Chlorobenzene	108-90-7
Chlorodibromomethane (Dibromochloromethane)	124-48-1
Chloroethane	75-00-3
Chloroform	67-66-3
Chloromethane	74-87-3
cis-1,2-Dichloroethene	156-59-2
cis-1,3-Dichloropropene	10061-01-5
Dichlorodifluoromethane	75-71-8
Ethylbenzene	100-41-4
Methylene chloride (DCM)	75-09-2
o-Xylene	95-47-6
Styrene	100-42-5
Tetrachloroethene	127-18-4
Toluene	108-88-3
trans-1,2-Dichloroethene	156-60-5
trans-1,3-Dichloropropene	10061-02-6
Trichloroethene (Trichloroethylene)	79-01-6
Trichlorofluoromethane	75-69-4
Vinyl chloride	75-01-4

Analytes

6 More Analytes are Common to at least 5 Methods

Analyte	CAS	624.1	6200	524.2 4.1	8260D	CLP Low Med	CLP Trace
1,2,4-Trichlorobenzene	120-82-1		1	1	1	1	1
2-Butanone (MEK)	78-93-3	1		1	1	1	1
Acetone	67-64-1	1		1	1	1	1
Carbon disulfide	75-15-0	1		1	1	1	1
Isopropylbenzene	98-82-8		1	1	1	1	1
Methyl tert-butyl ether (MTBE)	1634-04-4		1	1	1	1	1

Analytes

17 More Analytes are Common to at least 4 Methods

Analyte	CAS	624.1	6200	524.2 4.1	8260D	CLP Low Med	CLP Trace
1,1,1,2-Tetrachloroethane	630-20-6	1	1	1	1		
1,2,3-Trichloropropane	96-18-4	1	1	1	1		
1,2,4-Trimethylbenzene	95-63-6	1	1	1	1		
1,3,5-Trimethylbenzene	108-67-8	1	1	1	1		
1,3-Dichloropropane	142-28-9	1	1	1	1		
2-Chlorotoluene	95-49-8	1	1	1	1		
4-Chlorotoluene	106-43-4	1	1	1	1		
Bromobenzene	108-86-1	1	1	1	1		
Dibromomethane	74-95-3	1	1	1	1		
m-Xylene	108-38-3	1	1	1	1		
n-Butylbenzene	104-51-8	1	1	1	1		
n-Propylbenzene	103-65-1	1	1	1	1		
p-Xylene	106-42-3	1	1	1	1		
sec-Butylbenzene	135-98-8	1	1	1	1		
tert-Butylbenzene	98-06-6	1	1	1	1		
2-Hexanone	591-78-6			1	1	1	1
4-Methyl-2-pentanone (MIBK)	108-10-1			1	1	1	1

Analytes

15 More Analytes are Common to at least 3 Methods

Analyte	CAS	624.1	6200	524.2 4.1	8260D	CLP Low Med	CLP Trace
1,1-Dichloropropene	563-58-6		1	1	1		
2,2-Dichloropropane	590-20-7		1	1	1		
4-Isopropyltoluene	99-87-6		1	1	1		
Hexachlorobutadiene	87-68-3		1	1	1		
Naphthalene	91-20-3		1	1	1		
1-Chlorobutane	109-69-3	1		1	1		
Acrylonitrile	107-13-1	1		1	1		
Allyl chloride	107-05-1	1		1	1		
Propionitrile (ethyl cyanide)	107-12-0	1		1	1		
trans-1,4-Dichloro-2-butene	110-57-6	1		1	1		
m+p- Xylene	179601-23-1	1				1	1
Cyclohexane	110-82-7				1	1	1
Methyl acetate	79-20-9				1	1	1
Methylcyclohexane	108-87-2				1	1	1
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1				1	1	1

Analytes

33 More Analytes are Common to at Least 2 of the 3 Most Common Methods (624.1, 524.2, 8260D)

Analyte	CAS	624.1	6200	524.2 4.1	8260D	CLP Low Med	CLP Trace
1,2,3-Trimethylbenzene	526-73-8	1			1		
2-Chloroethanol	107-07-3	1			1		
2-Chloroethyl vinyl ether	110-75-8	1			1		
2-Nitropropane	79-46-9			1	1		
Acetonitrile	75-05-8	1			1		
Acrolein (Propenal)	107-02-8	1			1		
Allyl alcohol	107-18-6	1			1		
Benzyl chloride	100-44-7	1			1		
Bromoacetone	598-31-2	1			1		
Chloral hydrate	302-17-0	1			1		
Chloroprene (2-chloro-1,3-butadiene)	126-99-8	1			1		
cis-1,4-Dichloro-2-butene	1476-11-5	1			1		
Crotonaldehyde	123-73-9	1			1		
Diethyl ether	60-29-7			1	1		
Ethyl methacrylate	97-63-2			1	1		
Ethyl t-butyl ether (ETBE)	637-92-3	1			1		
Hexachloroethane	67-72-1			1	1		
Iodomethane (Methyl iodide)	74-88-4			1	1		
Methacrylonitrile	126-98-7			1	1		
Methyl acrylate	96-33-3			1	1		
Methylmethacrylate	80-62-6			1	1		
n-Butanol (1-Butanol, n-Butyl alcohol)	71-36-3	1			1		
Nitrobenzene (NB)	98-95-3			1	1		
n-Propylamine	107-10-8	1			1		
o-Toluidine	95-53-4	1			1		
Pentachloroethane	76-01-7			1	1		
Pyridine	110-86-1	1			1		
t-Amyl ethyl ether (TAEE, 4,4-Dimethyl-3-oxahexane)	919-94-8	1			1		
t-Amyl methyl ether (TAME)	994-05-8	1			1		
t-Butyl alcohol (TBA)	75-65-0	1			1		
Vinyl acetate	108-05-4	1			1		
Chloroacetonitrile	107-14-2	1		1			
Tetrahydrofuran	109-99-9	1		1			

Analytes

The FINAL 30 are unique to only 1 Method

Analyte	CAS	624.1	6200	524.2 4.1	8260D	CLP Low Med	CLP Trace
1,2,3,4-Diepoxybutane	1464-53-5				1		
1,3-Dichloro-2-propanol	96-23-1				1		
1,4-Dioxane	123-91-1				1		
1-Chlorohexane	544-10-5				1		
1-Propanol (n-Propyl alcohol)	71-23-8				1		
2-Pentanone	107-87-9				1		
2-Picoline (2-Methylpyridine)	109-06-8				1		
2-Propanol (Isopropyl alcohol)	67-63-0				1		
Diisopropyl ether (DIPE)	108-20-3				1		
Epichlorohydrin	106-89-8				1		
Ethanol	64-17-5				1		
Ethyl acetate	141-78-6				1		
Ethylene oxide	75-21-8				1		
Isobutyl alcohol	78-83-1				1		
Malononitrile	109-77-3				1		
Methanol	67-56-1				1		
N-Nitroso-di-n-butylamine (N-Nitrosodibutylamine)	924-16-3				1		
Paraldehyde	123-63-7				1		
Pentafluorobenzene	363-72-4				1		
Propargyl alcohol	107-19-7				1		
β-Propiolactone	57-57-8				1		
1,1-Dichloropropanone (1,1-Dichloro-2-propanone)	513-88-2			1			
1,3-Butadiene	106-99-0	1					
1-Chlorohexanone	20261-68-1	1					
3-Chloropropionitrile	542-76-7	1					
bis (2-Chloroethyl) sulfide	505-60-2	1					
Chlorodifluoromethane	75-45-6	1					
Cyclohexanone	108-94-1	1					
m+o- Xylene	179601-22-0	1					
o+p- Xylene	136777-61-2	1					

Similarities & Coverage

More similarities than differences……..

Summary of Comparison

- Methods were developed by the individual programs to meet the needs of that program……but there are numerous targets that are common to all programs
- Enough similarities exist to form a good basis for a single method
- The comparison of collection and handling requirements show that the methods have enough similarity that a best practice could easily be determined
- The differences could easily be addressed by sample type:
- Common sense would disallow using the method for the analysis of a trace or DW sample and known contaminated samples in the same analytical run

Conclusion

There is enough interest to support further exploration……..

FUTURE FOCUS
• Fit for Use ✓Flexibility to meet compliance needs ✓Sensitivity ✓Technology ✓Quality

Questions?

Thank you!