ASTM D7575
‘Green’ Solventless Infrared Oil and Grease Method

Orono Spectral Solutions

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USEPA Visit to OSS 2/23/11

OIL AND GREASE MEASUREMENT SUMMARY

OIL AND GREASE MEASUREMENT

OIL AND GREASE MEASUREMENT:

- One of the five conventional pollutants covered by the 1974 Clean Water Act
- Second most-enforced-against parameter, second only to pH.
- Measurement included in all: NPDES permits, pre-treatment permits, Industrial Effluent Guidelines.
- **Millions of oil and grease analyses each year in the US alone**.

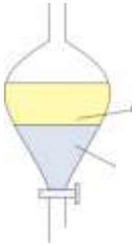
MEASUREMENT HISTORY:

- Montreal Protocol in 1989
- EPA moved from a Freon extraction method (EPA 413) to a n-Hexane extraction mass-based determination method (EPA 1664 in ‘95 / EPA 1664a in ’99 allowing LLE and SPE **=CURRENT METHOD**)
- ISSUES:
    - personal exposure, handling, and transportation of a hazardous, flammable liquid is required,
    - n-Hexane is a known neurotoxin,
    - analytical time is significantly increased, and
    - millions of liters/year of *n*-Hexane (not to mention other method solvents) for disposal.
    - methodology inconsistent with the intent of the Clean Air Act and Clean Water Act that considers *n*-Hexane a hazardous pollutant.
    - ASTM D7575 ‘solid phase infrared amenable extractor’ technology will remedy the issues listed above by eliminating solvents from oil and grease analysis – all while providing a more economical and accurate solution.

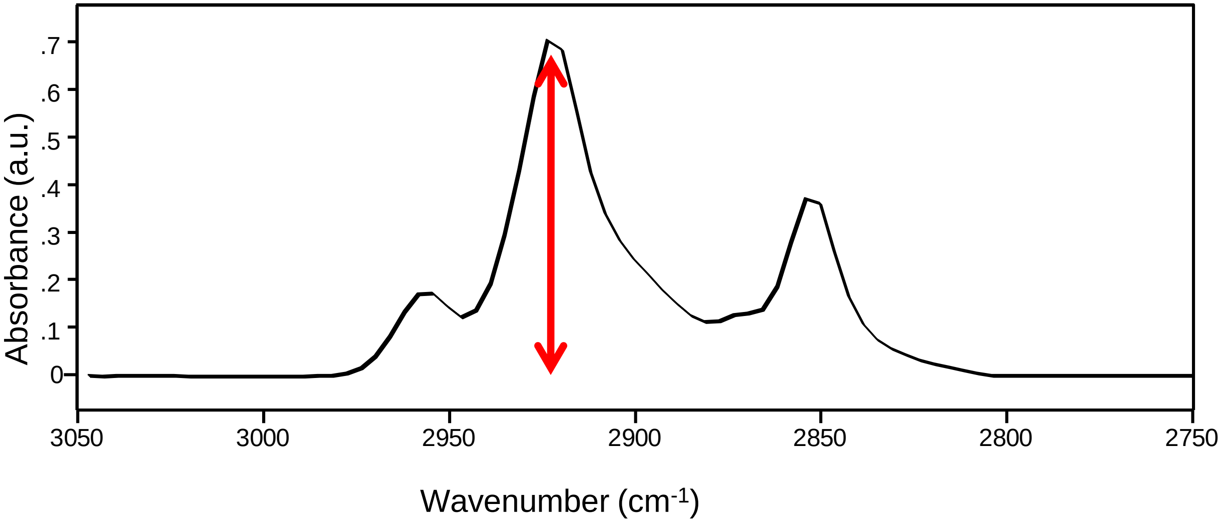
Oil and Grease Method Evolution

LLE

EPA 413.2 (Freon Extraction - Infrared)



Cuvette in Spectrometer



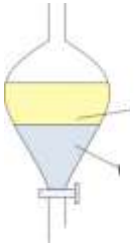
Peak height corresponds to hydrocarbon concentration

Time for analysis ~ minutes

Oil and Grease Method Evolution

LLE

EPA 1664



Evaporate Hexane

Mass corresponds to concentration of extractables

Time ~ 3 - 4 hours

n-Hexane use introduces four issues:

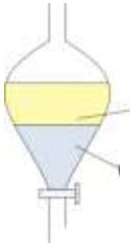
1. Flammable solvent
2. Known neurotoxin
3. Analytical time increased significantly
4. Large amount of solvent for disposal to atmosphere or hazardous waste

Total Hexane Usage: Millions of Liters/year

Oil and Grease Method Evolution

EPA 1664A

LLE



↓

Extract in drying pan

↓

Evaporate Hexane

Mass corresponds to concentration of extractables

Time ~ 3-4 hours

SPE

1. Sample run through SPE disk
2. Disk Eluted with Hexane

↓

Extract in drying pan

Evaporate Hexane

EPA allows SPE Option for 1664 without prior demonstration of equivalency! (Because of advantages in solvent reduction, etc.)

Federal Register /Vol. 64, No. 93 / Friday, May 14, 1999 /Rules and Regulations 26317

C. Use of Solid-Phase Extraction (SPE)

…A detailed description of the SPE technique was provided at proposal (61 FR 1730). Even prior to proposal of Method 1664, vendors of SPE devices had requested that SPE be an allowed technique in the Method. Proposed Method 1664 allowed use of SPE, but required a demonstration that SPE produced results equivalent to results produced by the separatory funnel liquid-liquid extraction technique (LLE) written in Method 1664. Vendors and other commenters objected to this requirement, claiming that **SPE provided sufficient advantages in solvent reduction, reduced analysis time, reduced emulsion formation, and other advantages so that its use should be allowed without prior demonstration of equivalency.** EPA discussed the issue extensively at proposal and in public workshops and meetings, and specifically solicited data demonstrating equivalency of results produced by SPE and LLE. Data received were mixed, with some data demonstrating that results produced are equivalent and other data demonstrating that results produced are significantly different. EPA reopened the comment period (61 FR 26149) to allow submission of further data, and EPA provided a notice of availability (62 FR 51621) of these and other data so that EPA could consider these data for today's final rule.

Discussions of the detailed issues on SPE are summarized in Section VI of this preamble and given in the detailed comments and responses included in the Docket. Based on comments received and supporting data, **EPA is allowing the use of SPE in the version of Method 1664 being approved today without a prior demonstration of equivalency.** However, EPA has added a note at the beginning of the extraction procedure (Section 11.3) in Method 1664 to indicate that it is the discharger/ generator's responsibility to assure that the results produced are equivalent. If there is doubt about this equivalency, liquid/liquid extraction is definitive for the measurement.

EPA also acknowledges that if a Region, State, or other permitting authority has concerns about the difference in results produced by SPE and LLE, that authority may specify in the permit the use of one of the two techniques

Oil and Grease Method Evolution

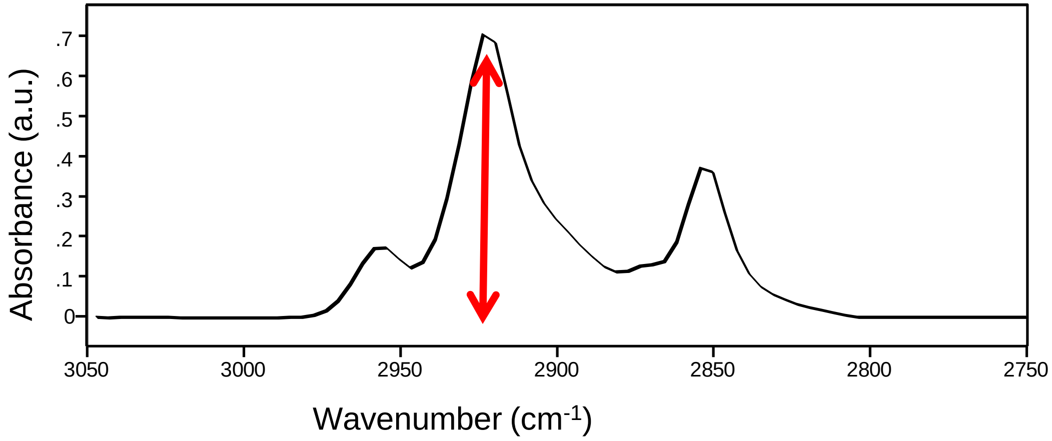
<u>New OSS Method:</u> **ASTM D7575 – Standard Test Method for Solvent-Free Membrane Recoverable Oil and Grease by Infrared Determination**

Sample run through **new IR-amenable oil and grease Extractor** created by OSS

Extractor placed in spectrometer

Peak height corresponds to hydrocarbon concentration

<u>Time ~ minutes</u>



ASTM D7575 VALIDATION

ASTM D7575 DEVELOPMENT MILESTONES

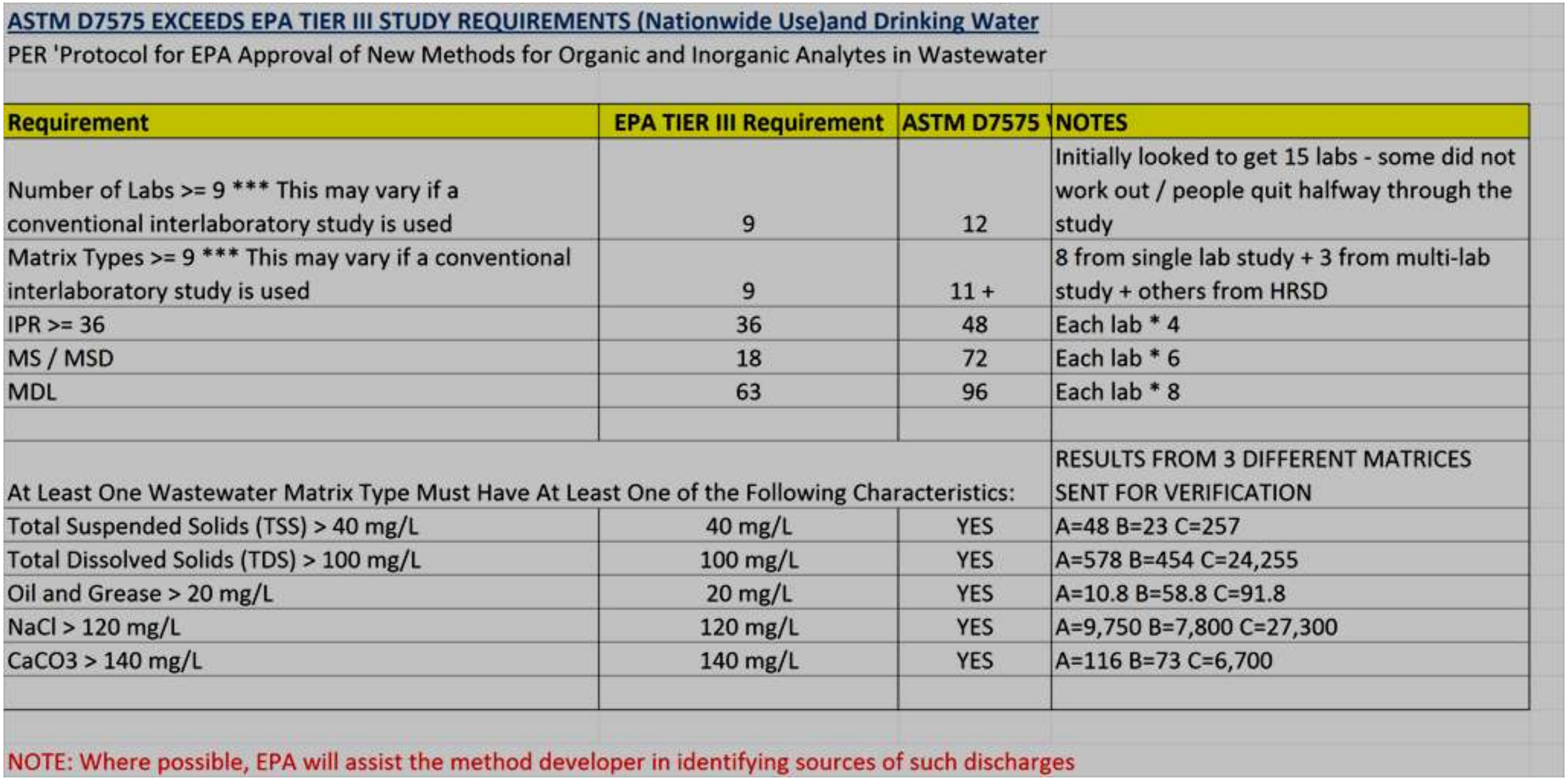
- Initial ASTM D19 committee presentation of technology Denver, CO (June 2008)
- Updated presentation of technology Cocoa Beach, FL (January 2009)
- USEPA presentation to Dick Reding, Lem Walker, Marion Kelly, Richard Englert, Richard Engler (Green Chemistry)
  - When Dick was asked specifically about his thoughts on the OSS method and data he commented:
    - Anything that gets rid of use of separatory funnels is a winner
    - Pluses: no solvent/co-solvent issues, good for environment, good environmental argument to move the method forward, ASTM approval will be a bonus.
    - Difficulties that we may be up against – no one wanting to change, issues with current method, another method defined parameter
    - Pull from the Green Chemistry office could be a key to helping this move forward – with a key point of a new method being solventless.
  - When asked about potential inclusion in the update rule Dick commented:
    - He would be comfortable putting this method out for comment
    - Finishing the single-lab validation study will be a good starting point
    - Completion of a multi-lab would not be required in order to get into the update rule – however – a multi-lab plan would be helpful
  - There was general agreement around the table that the matrices we have tested or are planning to test encompassed a sufficiently broad range of matrices.
- ASTM single lab validation study – technology gives same results as current EPA 1664 method with 95% confidence across spectrum of wastewater matrices (June 2009)
- Present single lab results at ASTM D19 Meeting in Las Vegas (June 2009)
  - single lab results approved
  - Multilab study plan approved by EPA (Reding, Walker) and ASTM D19.06 members
- ASTM multilaboratory validation study – 12 independent labs (August 2009)
- ASTM D7575 method assignment (December 2009)
- ASTM D7575-10 Final Approval (January 2010)

ASTM D7575 Validation

- New technology and method validated through ASTM process per direction of Dick Reding (ret., Dir. EAD-OST-OW-EPA at the time)
- Two step process:
  - Single lab validation to show preliminary precision as well as comparability to 1664
  - Multi-lab validation to show precision across laboratories and establish QC limits

ASTM D7575 Development **Exceeds** EPA Tier III Requirements

(for NATIONWIDE use)



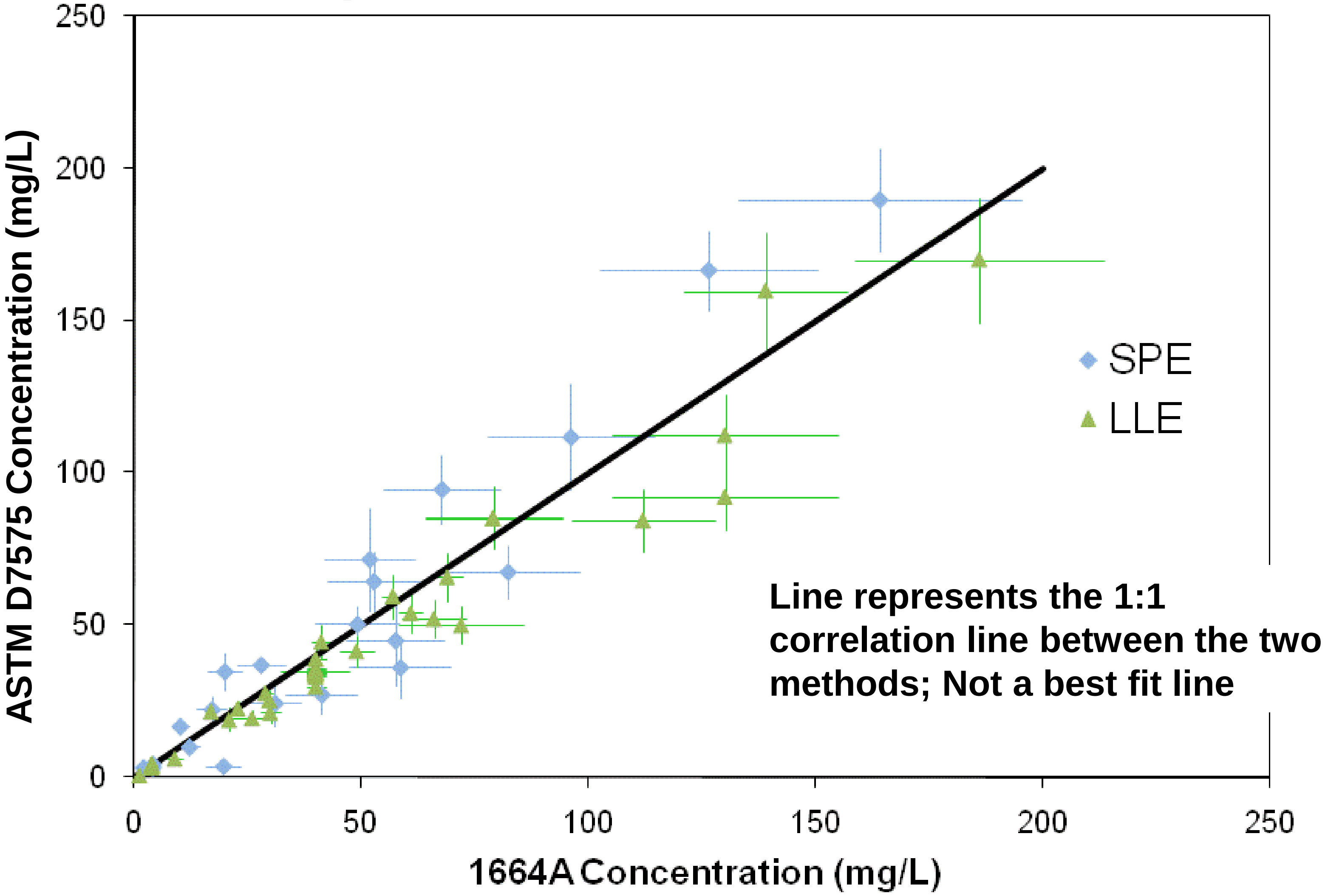
ASTM D7575 EXCEEDS EPA TIER III STUDY REQUIREMENTS (Nationwide Use)and Drinking Water

PER 'Protocol for EPA Approval of New Methods for Organic and Inorganic Analytes in Wastewater

Requirement	EPA TIER III Requirement	ASTM D7575	NOTES
Number of Labs >= 9 *** This may vary if a conventional interlaboratory study is used	9	12	Initially looked to get 15 labs - some did not work out / people quit halfway through the study
Matrix Types >= 9 *** This may vary if a conventional interlaboratory study is used	9	11 +	8 from single lab study + 3 from multi-lab study + others from HRSD
IPR >= 36	36	48	Each lab * 4
MS / MSD	18	72	Each lab * 6
MDL	63	96	Each lab * 8
At Least One Wastewater Matrix Type Must Have At Least One of the Following Characteristics:			RESULTS FROM 3 DIFFERENT MATRICES SENT FOR VERIFICATION
Total Suspended Solids (TSS) > 40 mg/L	40 mg/L	YES	A=48 B=23 C=257
Total Dissolved Solids (TDS) > 100 mg/L	100 mg/L	YES	A=578 B=454 C=24,255
Oil and Grease > 20 mg/L	20 mg/L	YES	A=10.8 B=58.8 C=91.8
NaCl > 120 mg/L	120 mg/L	YES	A=9,750 B=7,800 C=27,300
CaCO3 > 140 mg/L	140 mg/L	YES	A=116 B=73 C=6,700

NOTE: Where possible, EPA will assist the method developer in identifying sources of such discharges

Comparison to EPA 1664



Line represents the 1:1 correlation line between the two methods; Not a best fit line

(OSS method tested by OSS, 1664A tested at independent lab)

Matrices

Tested During Development

Set 1		Set 2		Set 3		Set 4
Gulf A		Stormwater Runoff		Cleaning Co.		POTW
Gulf B		Food Processor A		Bakery		Dairy
Food Processor		Food Processor B		Prison		Fish Processor
Prison		Food Processor C		Hospital A		Auto Salvage
POTW - Primary Effluent		POTW - Primary Effluent		Hospital B		Auto Garage
Paper Mill		Prison		Hospital C		Machinist
		Paper Mill		Hospital D		Gunsmith
				Hospital E		
				Gunsmith		
				Stormwater Runoff		

(Tested by OSS, compared to 1664A, LLE and/or SPE)

Single Lab Validation

- Completed Single Lab Validation – (June ‘09)
- Performed at independent lab
- Key purpose:
  - Method Comparability
- Compared Results to EPA 1664
- Balloted ASTM Single-Lab Validated Method

Single Lab Validation Data

(2 Matrices, 7 Replicates)

	Measured Result in mg/L for Real-World Matrices	
Replicate #	**Publicly Owned Treatment Works**	**Gunsmith**
1	31.3	76.1
2	29.2	73.3
3	24.7	75.3
4	30.7	78.3
5	34.1	74.5
6	29.7	91.2
7	27.4	89.4
Average Recovery (mg/L)	**29.6**	**79.7**
Average Recovery by EPA 1664 (mg/L)	**32.2**	**78.9**
Average Recovery as % of EPA 1664	92%	101%
Standard Deviation (mg/L)	3.0	7.4
Relative Standard Deviation as %	10%	9.3%

Single Lab Validation Data (cont.)

(5 Matrices, 3 Replicates)

Measured Result mg/L as Oil and Grease					
Replicate #	**Auto Garage**	**Dairy**	**Machine Shop**	**Auto Salvage Yard**	**Fish Processor**
1	21.0	89.5	74.0	5.8	50.5
2	19.9	91.4	69.9	4.8	54.1
3	18.4	103.8	98.4	5.8	50.9
Average Recovery (mg/L)	**19.8**	**94.9**	**80.8**	**5.5**	**51.8**
Average Recovery by EPA 1664 (mg/L)	**21.1**	**108.8**	**89.4**	**6.6**	**54**
Average Recovery as % of EPA 1664	94%	87%	90%	84%	96%
Standard Deviation (mg/L)	1.3	7.8	15.4	0.6	2.0
Relative Standard Deviation as %	6.7%	8.2%	19%	10%	3.8%
Matrix Spike Recovery %	101%	**	96%	94%	94%

** Not tested

Single Lab Validation Data (cont.)

(ASTM D7575 vs. EPA 1664 Average Recovery = 96.2%)

Seven samples of one matrix over 1 month period: POTW

Measured Result mg/L as Oil and Grease							
Replicate #	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7
1	22.6	25.6	21.7	23.2	19.6	28.8	1.1*
2	19.5	29.9	14.8	20.8	19.5	25.7	1.3*
3	23.7	28.6	21.2	25.1	25.9	22.6	0.9*
Average Recovery (mg/L)	**21.9**	**28.0**	**19.3**	**23.0**	**21.7**	**25.7**	**1.1***
Average Recovery by EPA 1664 (mg/L)	**17.0**	**28.9**	**20.9**	**22.9**	**30.0**	**29.8**	**1.2***
Average Recovery as % of EPA 1664	129%	97%	92%	101%	72%	86%	N/A**
Standard Deviation (mg/L)	2.2	2.2	3.8	2.1	3.7	3.1	N/A**
Relative Standard Deviation, %	10%	8%	20%	9%	17%	12%	N/A**
Matrix Spike Recovery %	96%	96%	88%	***	***	***	110%

*   Below recommended reporting limit

** Not calculated – date below recommended reporting limit

*** Not performed

Single Lab Validation Summary

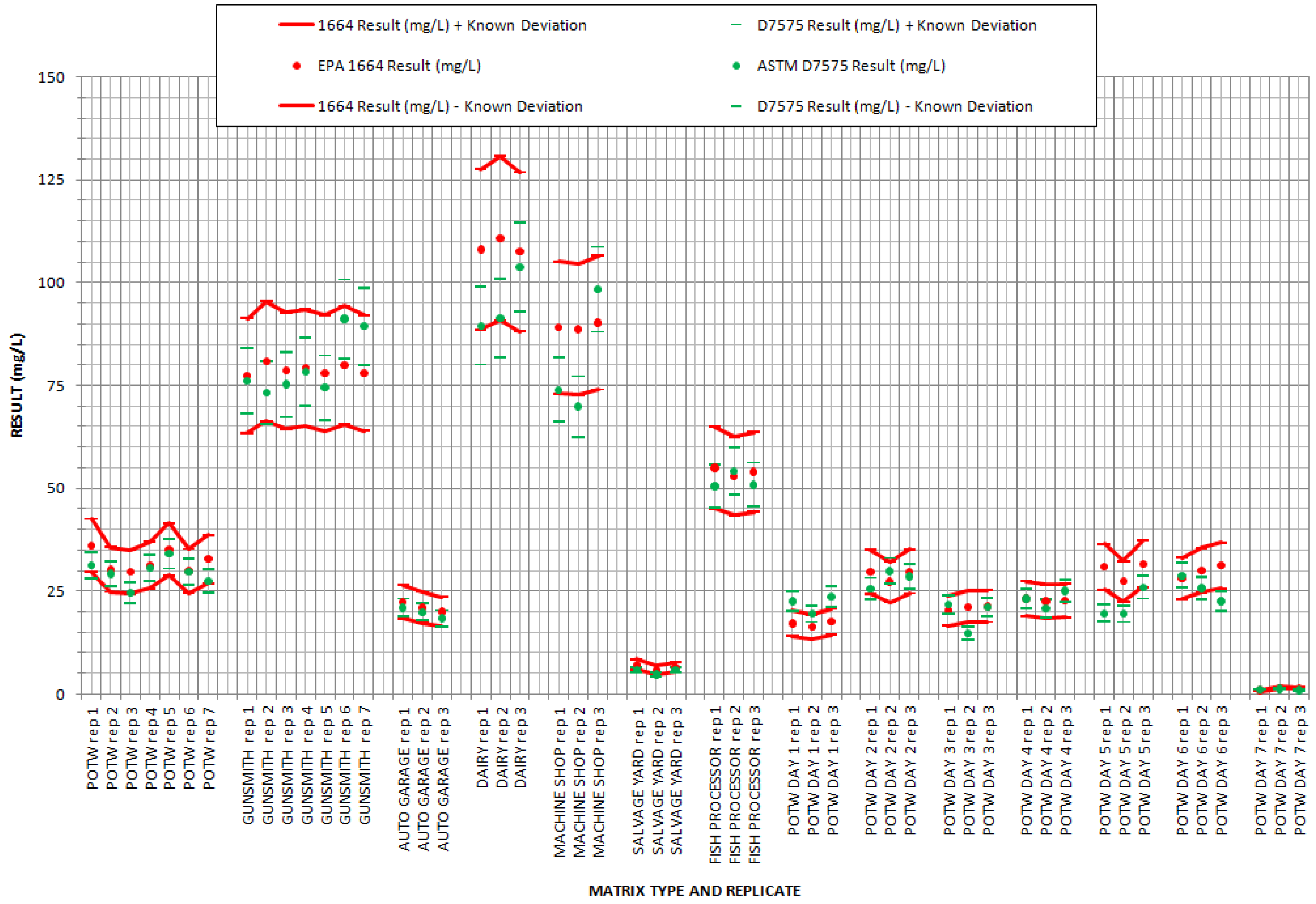
- EPA Method Comparability Statistics
  - NO DIFFERENCE IN RESULTS at 95% CONFIDENCE!

* ASTM E178 Outliers Removed

SINGLE LAB METHOD COMPARABILITY RESULTS

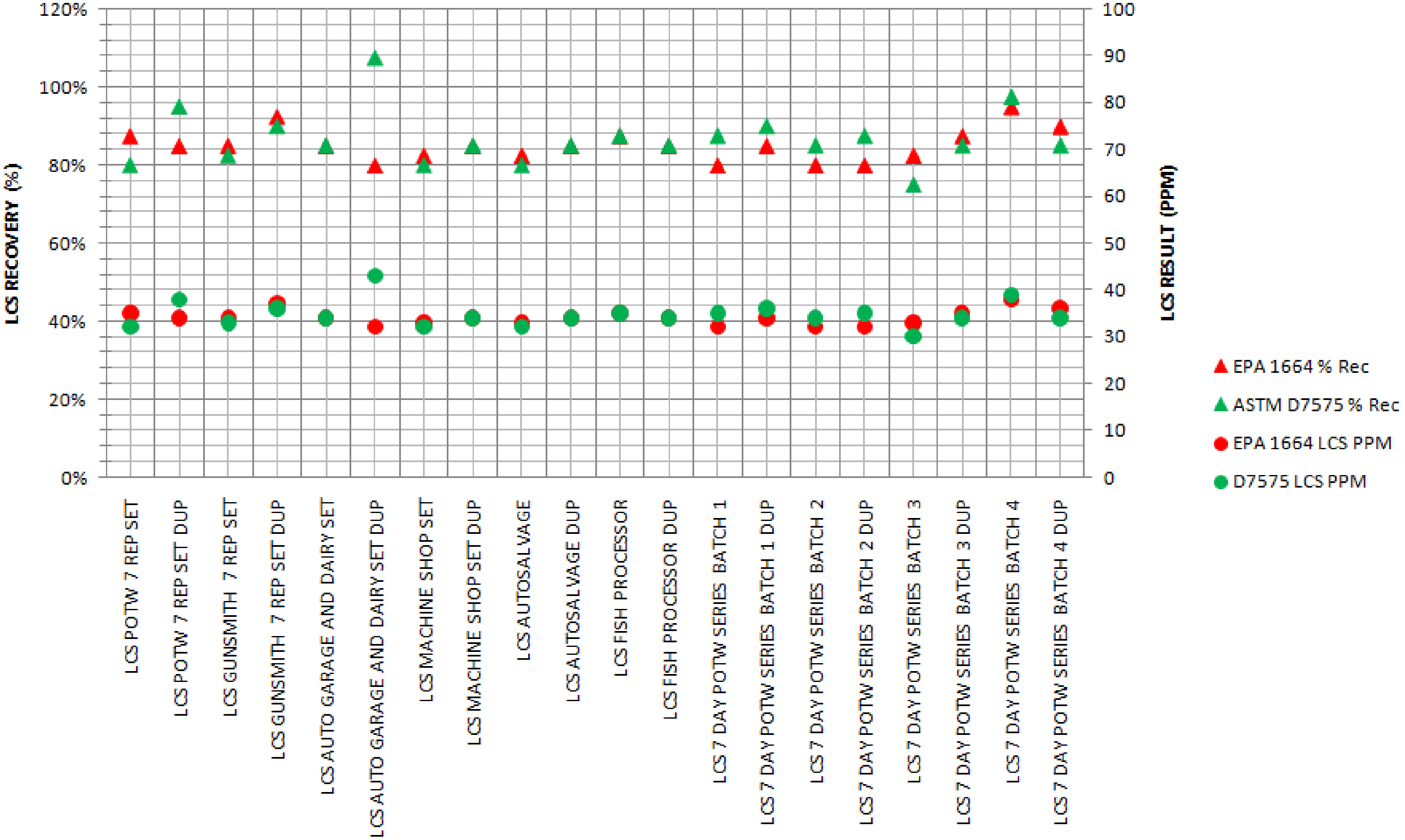
SINGLE LAB STUDY: D7575 vs. EPA 1664 Results (showing known deviations)

NOTE: DATA RANGES OVERLAP!



ASTM D7575 vs EPA 1664 Comparative Study Laboratory Control Samples (Equivalent Recovery Performance!)

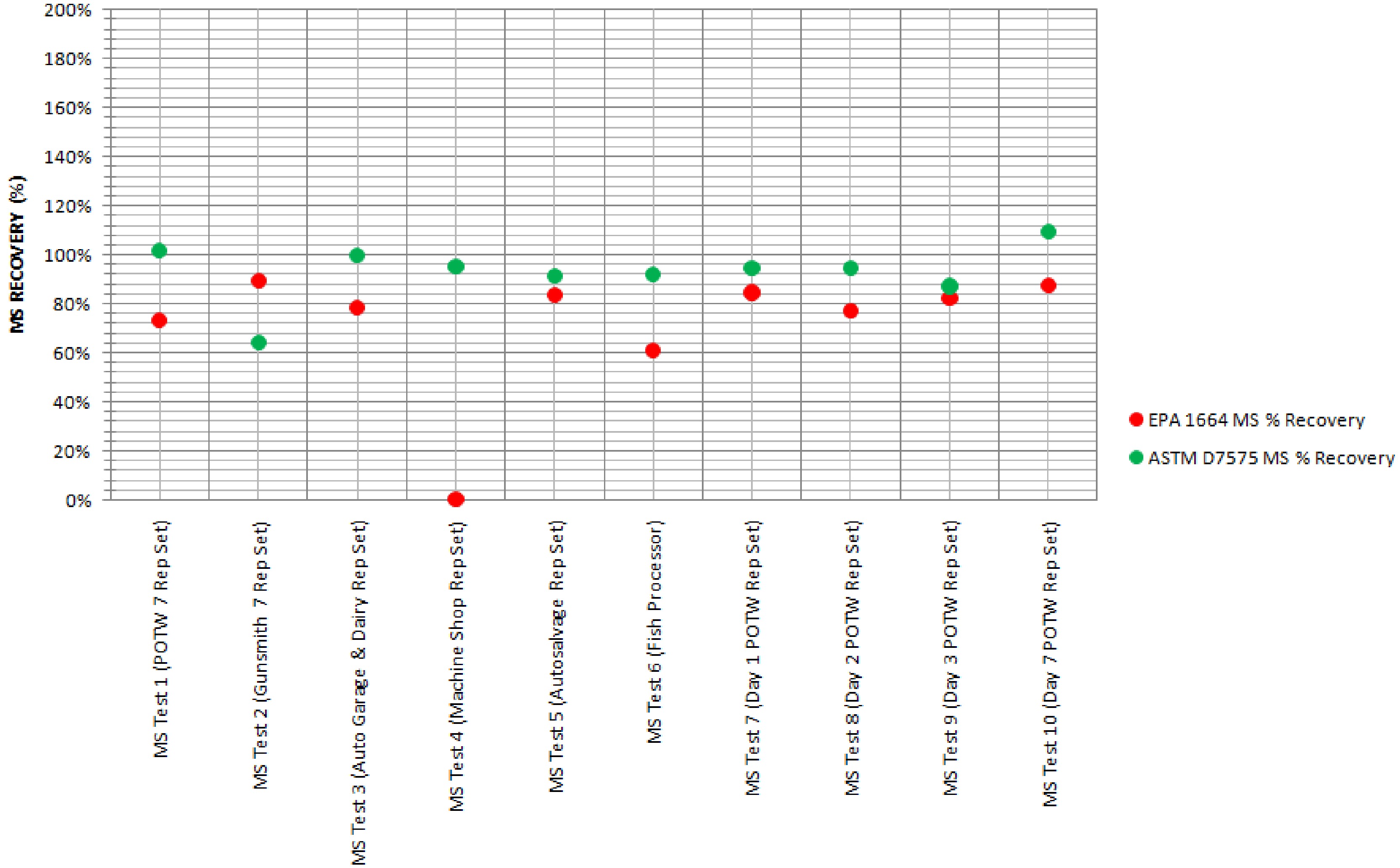
ASTM D7575 (ave=87% recovery)
EPA 1664 (ave=85% recovery)



ASTM D7575 vs EPA 1664 Comparative Study Matrix Spike Recovery

(ASTM D7575 Outperforms EPA 1664!)

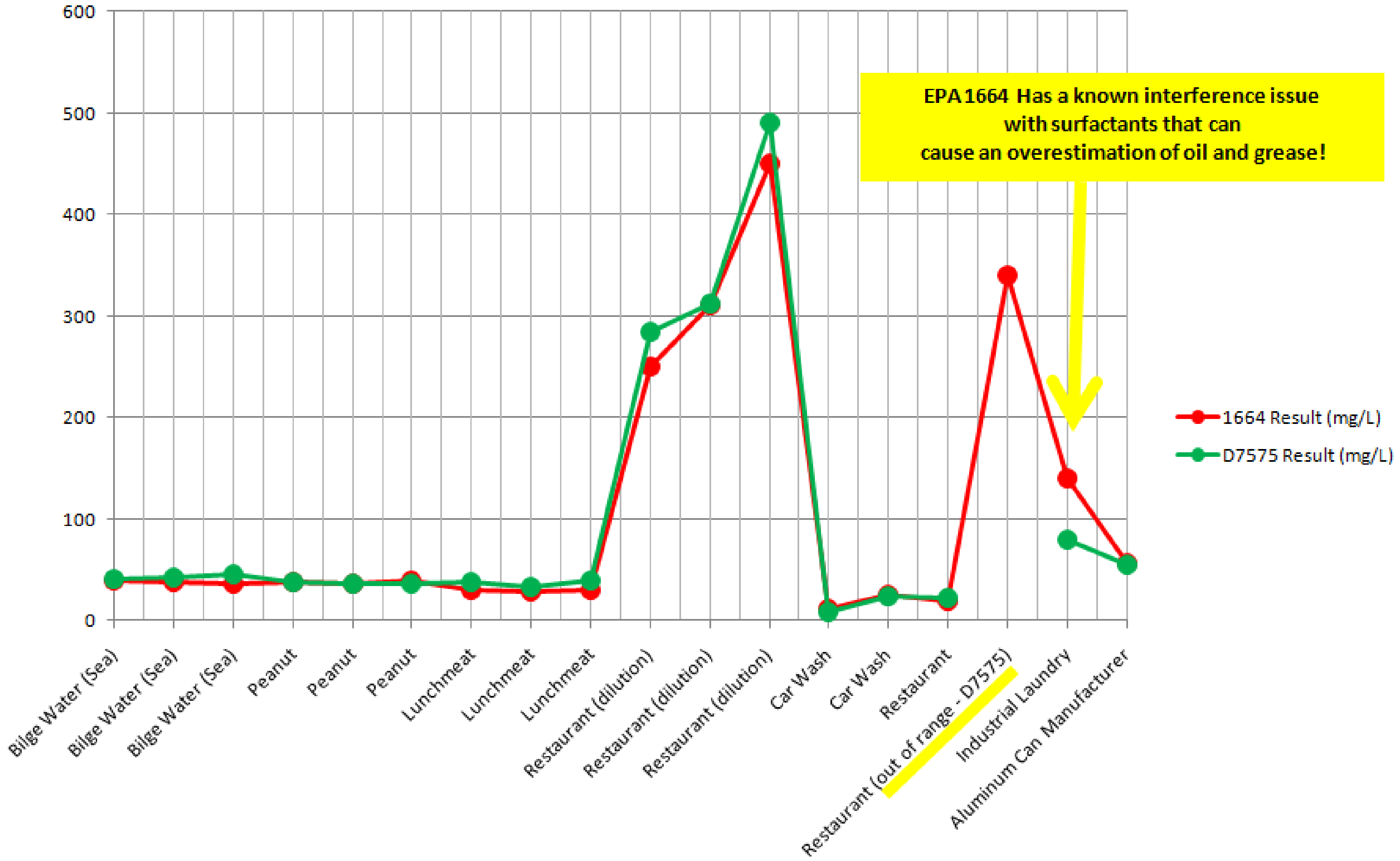
ASTM D7575 (ave=94% recovery) >> EPA 1664 (ave=71% recovery)(ave=78% w/ outlier removed)



ASTM D7575 VS EPA 1664 COMPARISON ACROSS MORE MATRICES

(PER USEPA REQUEST FOR INFORMATION OVER AND ABOVE ASTM VALIDATION DATA)

(some triplicate and some single test results)



EPA 1664 Has a known interference issue
with surfactants that can
cause an overestimation of oil and grease!

Supplemental Data and Statistical Analysis in Support of Method Equivalence of ASTM D7575 Solventless Oil and Grease and EPA Method 1664A

Barrett P. Eynon
Statistician

Introduction

I am a **professional statistician** with over **30 years of experience in applying statistics** to the environmental and biotechnology fields. In the early 1990s. I was a **statistical consultant to the US EPA Office of Water and provided statistical and data analyses of laboratory measurement data for Oil and Grease in the EPA Freon Replacement studies**, including the Phase 1(US EPA, 1993), Phase 2(US EPA, 1995) and Validation (US EPA, 1995) studies that **led to EPA Method 1664A** (US EPA, 2000), in work done through SRI International and Dyncorp/VIAR (EPA Sample Control Center).

Conclusion

The analysis presented here shows that the **ASTM D7575 Solventless Oil and Grease method is statistically equivalent to EPA Method 1664A** when considered in light of previous well-known precision data reported for EPA Method 1664A.

In my interest as a concerned citizen I would hope that that, given equivalence, EPA can provide users a solventless / greener alternative to the use of environmentally unfriendly and hazardous n-hexane as specified in Method 1664. Especially since the current solid-phase extraction version of EPA 1664 was adopted WITHOUT ANY comparability data because of the positive attributes it would provide (time savings, solvent use) over the liquid – liquid extraction procedure of EPA 1664.

MULTI-LAB TESTING

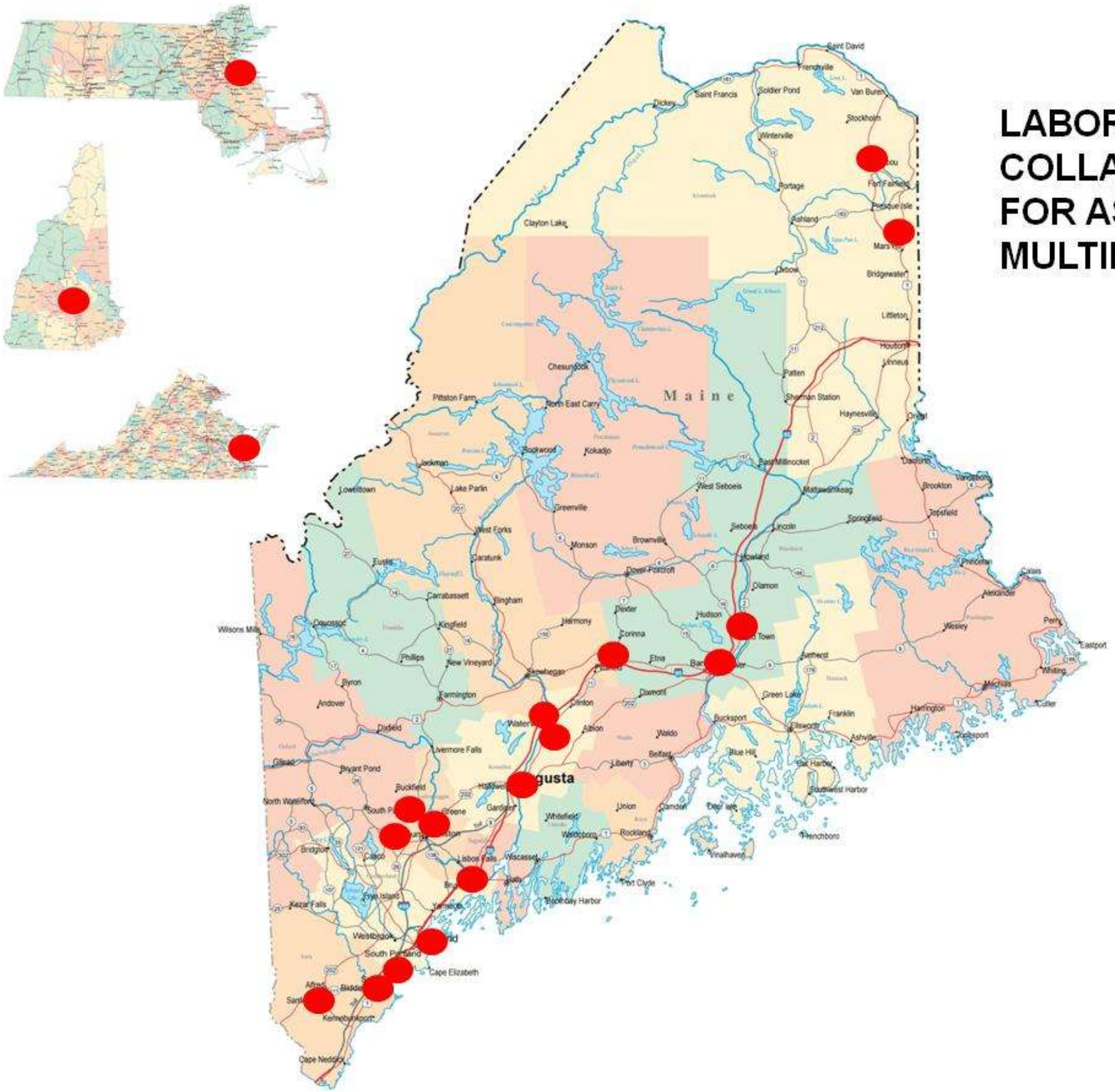
Multi-Lab Validation of ASTM D7575

- Key Purpose – Method Precision and Bias

- Completed – 12 Independent Labs (Aug./Sep. ‘09)

- Designed according to ASTM D2777
  - approved by ASTM D19.06 committee / EPA Office of Water personnel (Reding, Walker)

- Youden Pair analyses of three matrices - POTW (10ppm), DAIRY (60ppm), BILGE WATER (100ppm)



LABORATORIES AND COLLABORATORS FOR ASTM D7575 MULTILAB STUDIES

Round-Robin Results

Results\Matrix	POTW 1	POTW 2	Dairy 1	Dairy 2	Bilge 1	Bilge 2
True Value (mg/L)	**10.8**	**9.3**	**58.8**	**51.6**	**91.8**	**86.6**
# Sample Results	11	11	12	11	12	10
Average Recovery (mg/L)	**11.0**	**9.1**	**60.0**	**53.1**	**94.8**	**84.3**
Minimum Recovery (mg/L)	8.3	6.6	47.9	43.1	76.5	67.3
Maximum Recovery (mg/L)	13.6	12.4	68.7	62.8	107.1	95.3
Relative Overall Standard Deviation,%	16.8%	19.6%	11.6%	10.5%	10.2%	10.2%
Relative Single Operator Standard Deviation, %	16.5%		7.3%		5.2%	
# Matrix Spike Results	10	10	9	10	11	9
Average Matrix Spike Recovery %	109%	117%	91%	108%	87%	87%

(AVERAGE RECOVERIES = TRUE VALUES)

Round-Robin Results

Results\Matrix	**POTW 1**	**POTW 2**	**Dairy 1**	**Dairy 2**	**Bilge 1**	**Bilge 2**
True Value (mg/L)	10.8	9.3	58.8	51.6	91.8	86.6
# Sample Results	11	11	12	11	12	10
Average Recovery (mg/L)	11.0	9.1	60.0	53.1	94.8	84.3
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Maximum Recovery (mg/L)	13.6	12.4	68.7	62.8	107.1	95.3
Relative Overall Standard Deviation,%	**16.8%**	**19.6%**	**11.6%**	**10.5%**	**10.2%**	**10.2%**
Relative Single Operator Standard Deviation, %	16.5%		7.3%		5.2%	
# Matrix Spike Results	10	10	9	10	11	9
Average Matrix Spike Recovery %	109%	117%	91%	108%	87%	87%

(AVERAGE OVERALL RELATIVE STANDARD DEVIATION = 13.1%)

(WATER SAMPLE EXPERIENCED LABS = 9.2%)

Round-Robin Results

Results\Matrix	POTW 1	POTW 2	Dairy 1	Dairy 2	Bilge 1	Bilge 2
True Value (mg/L)	10.8	9.3	58.8	51.6	91.8	86.6
# Sample Results	11	11	12	11	12	10
Average Recovery (mg/L)	11.0	9.1	60.0	53.1	94.8	84.3
Minimum Recovery (mg/L)	8.3	6.6	47.9	43.1	76.5	67.3
Maximum Recovery (mg/L)	13.6	12.4	68.7	62.8	107.1	95.3
Relative Overall Standard Deviation,%	16.8%	19.6%	11.6%	10.5%	10.2%	10.2%
Relative Single Operator Standard Deviation, %	16.5%		7.3%		5.2%	
# Matrix Spike Results	10	10	9	10	11	9
Average Matrix Spike Recovery %	109%	117%	91%	108%	87%	87%

(AVERAGE SINGLE OPERATOR RELATIVE STANDARD DEVIATION = 9.7%)

(WATER SAMPLE EXPERIENCED LABS = 7.5%)

Round-Robin Results

Results\Matrix	POTW 1	POTW 2	Dairy 1	Dairy 2	Bilge 1	Bilge 2
True Value (mg/L)	10.8	9.3	58.8	51.6	91.8	86.6
# Sample Results	11	11	12	11	12	10
Average Recovery (mg/L)	11.0	9.1	60.0	53.1	94.8	84.3
Minimum Recovery (mg/L)	8.3	6.6	47.9	43.1	76.5	67.3
Maximum Recovery (mg/L)	13.6	12.4	68.7	62.8	107.1	95.3
Relative Overall Standard Deviation,%	16.8%	19.6%	11.6%	10.5%	10.2%	10.2%
Relative Single Operator Standard Deviation, %	16.5%		7.3%		5.2%	
# Matrix Spike Results	10	10	9	10	11	9
Average Matrix Spike Recovery %	**109%**	**117%**	**91%**	**108%**	**87%**	**87%**

(AVERAGE SPIKE RECOVERY = 99.8%)

Range / QC Acceptance Limits

Method Detection Limit and Reporting Range

Analyte	MDL (mg/L)	Reporting Range (mg/L)
Oil and Grease	1.0	5-200

QC Acceptance Criteria

Analyte	Initial Precision and Recovery				Lab Control Sample		Matrix Spike	
	Test Concentration (mg/L)	Recovery (%)		Precision	Recovery (%)		Recovery (%)	
		Lower limit	Upper limit	Maximum RSD (%)	Lower limit	Upper limit	Lower limit	Upper limit
Oil and Grease	40	88	105	10.5	79	113	69	127

All labs were able to meet QC performance spec. within 1 (most) or 2 attempts

Round-Robin Results

YOUDEN PAIR STATISTICS:

- Overall relative standard deviation (RSD) 13.1% (across all labs, samples, operators)
  - Water sample EXPERIENCED labs overall RSD 9.2%

- Within-lab Operator RSD 9.7% (across all samples)
  - Water sample EXPERIENCED labs within-lab RSD 7.5%

Round-Robin Results (MDL,ML)

(Method Detection Limit = 1.003 ppm)
(Method Limit = 3.181 ppm)

Parameter	MDL
Amount Spiked (mg/L)	4
# Results	12
Pooled MDL (mg/L)	**1.0**
Standard Deviation of MDL (mg/L)	*0.26*
Pooled Method Limit (mg/L)	**3.181***

*** NOTE: EPA 1664 Method Limit = 5 mg/L**

All labs were able to meet MDL performance spec. within 1 (most) or 2 attempts

Round-Robin Results (IPR)

(Average Recovery = 96%)

(95% Confidence Recovery Range Limits = 84% - 107%)

Parameter	***IPR***	
	Mean Recovery	***Precision***
Amount Spiked (mg/L)	*40*	*40*
# Results	*12*	*12*
Average Recovery%	***96%***	
Standard Deviation of Recovery	*5.07%*	
Average RSD		*7.53%*
Standard Deviation of RSD		2.24%

All labs were able to meet IPR performance spec. within 1 (most) or 2 attempts

Round-Robin Results (LCS)

(Average Recovery = 100%)

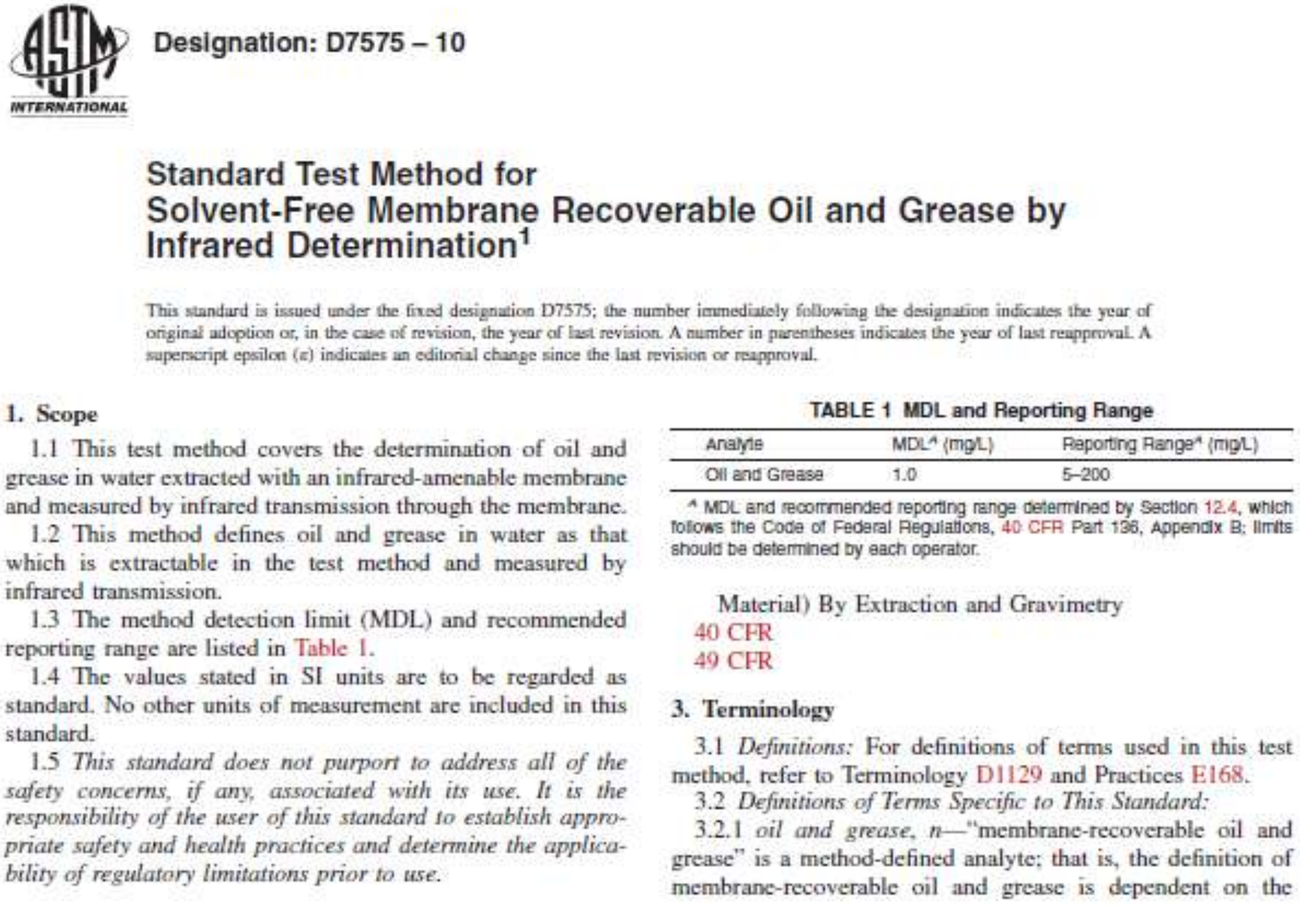
(95% Confidence Recovery Range Limits = 81% - 118%)

Parameter	LCS
Amount Spiked (mg/L)	40
# Results	12
Average %	**100%**
Standard Deviation of LCS (mg/L)	8.0%

All labs were able to meet LCS performance spec. within 1 (most) or 2 attempts

ASTM D7575

Published January 2010 and available for purchase at ASTM website



Designation: D7575 – 10

Standard Test Method for Solvent-Free Membrane Recoverable Oil and Grease by Infrared Determination[1]

This standard is issued under the fixed designation D7575; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of oil and grease in water extracted with an infrared-amenable membrane and measured by infrared transmission through the membrane.

1.2 This method defines oil and grease in water as that which is extractable in the test method and measured by infrared transmission.

1.3 The method detection limit (MDL) and recommended reporting range are listed in Table 1.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

TABLE 1 MDL and Reporting Range

Analyte	MDL[A] (mg/L)	Reporting Range[A] (mg/L)
Oil and Grease	1.0	5–200

[A] MDL and recommended reporting range determined by Section 12.4, which follows the Code of Federal Regulations, 40 CFR Part 136, Appendix B; limits should be determined by each operator.

Material) By Extraction and Gravimetry

40 CFR

49 CFR

3. Terminology

3.1 *Definitions:* For definitions of terms used in this test method, refer to Terminology D1129 and Practices E168.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *oil and grease, n*—"membrane-recoverable oil and grease" is a method-defined analyte; that is, the definition of membrane-recoverable oil and grease is dependent on the

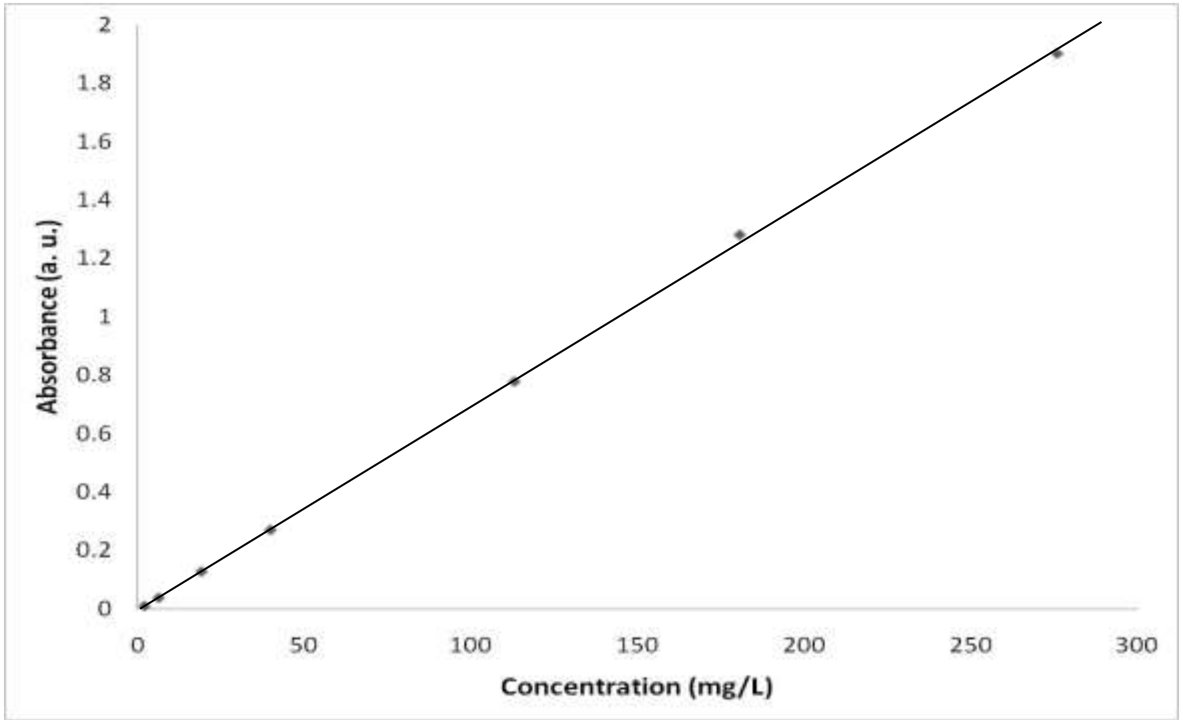
ASTM D7575 METHOD PROCESS

ASTM Method D7575 Video

Calibration Standard Devices

Calibration of infrared instrumentation performed with ‘Standard Devices’, which contain a known amount of material

SOLID STATE DEVICES – Nothing to make up!



Calibration Verification also performed daily using one of the Standard Devices

ASTM D7575 Method

Real Sample
-acidified
-cooled

→

1) Homogenize
(e.g. sonicate, mechanical shake, or hand shake)
(1-20 minutes)

→

2) Fill Sterile Disposable Syringe

→

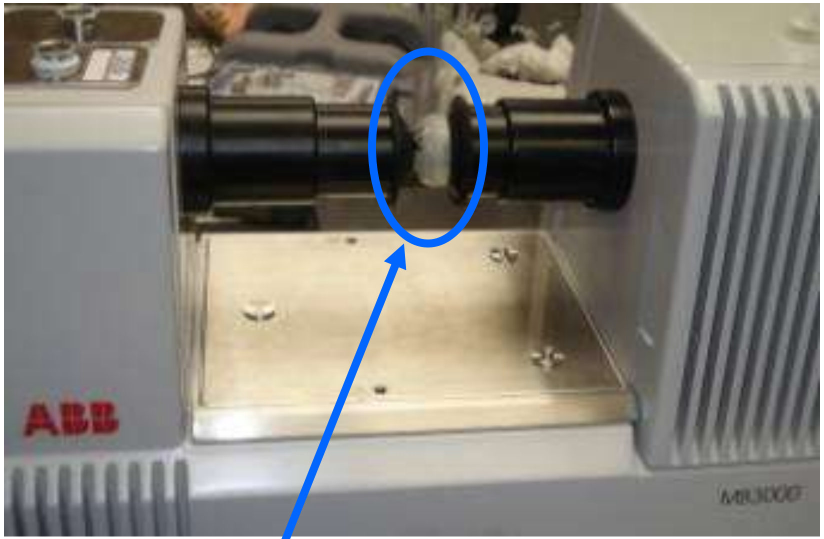
Method (con’t)

Attach OSS Device to Syringe

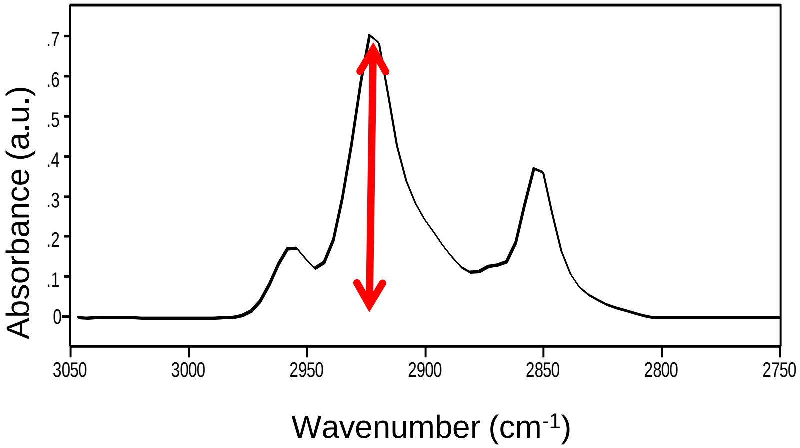
Use Syringe Pump to Force Sample through Device (3 minutes)

Use Air Pump to Aid Drying (2-45 minutes)

Method (con't)



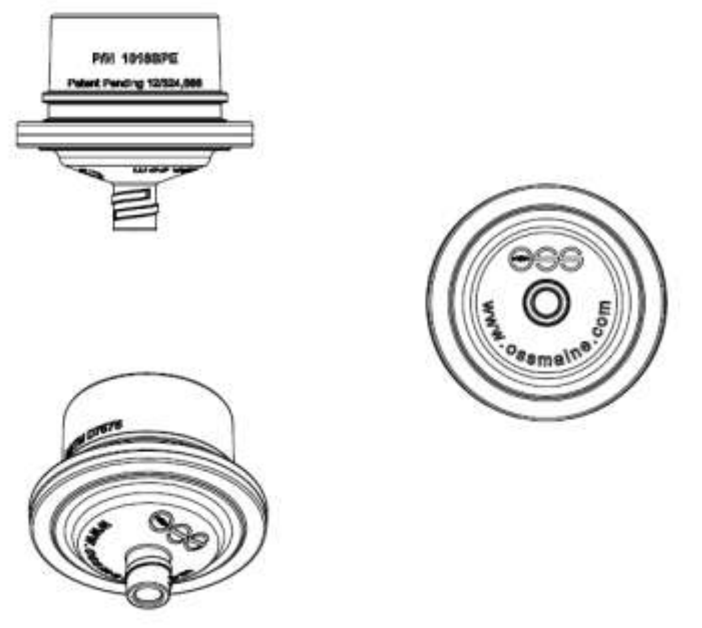
Examine with IR Spectrometer
(1 minute)



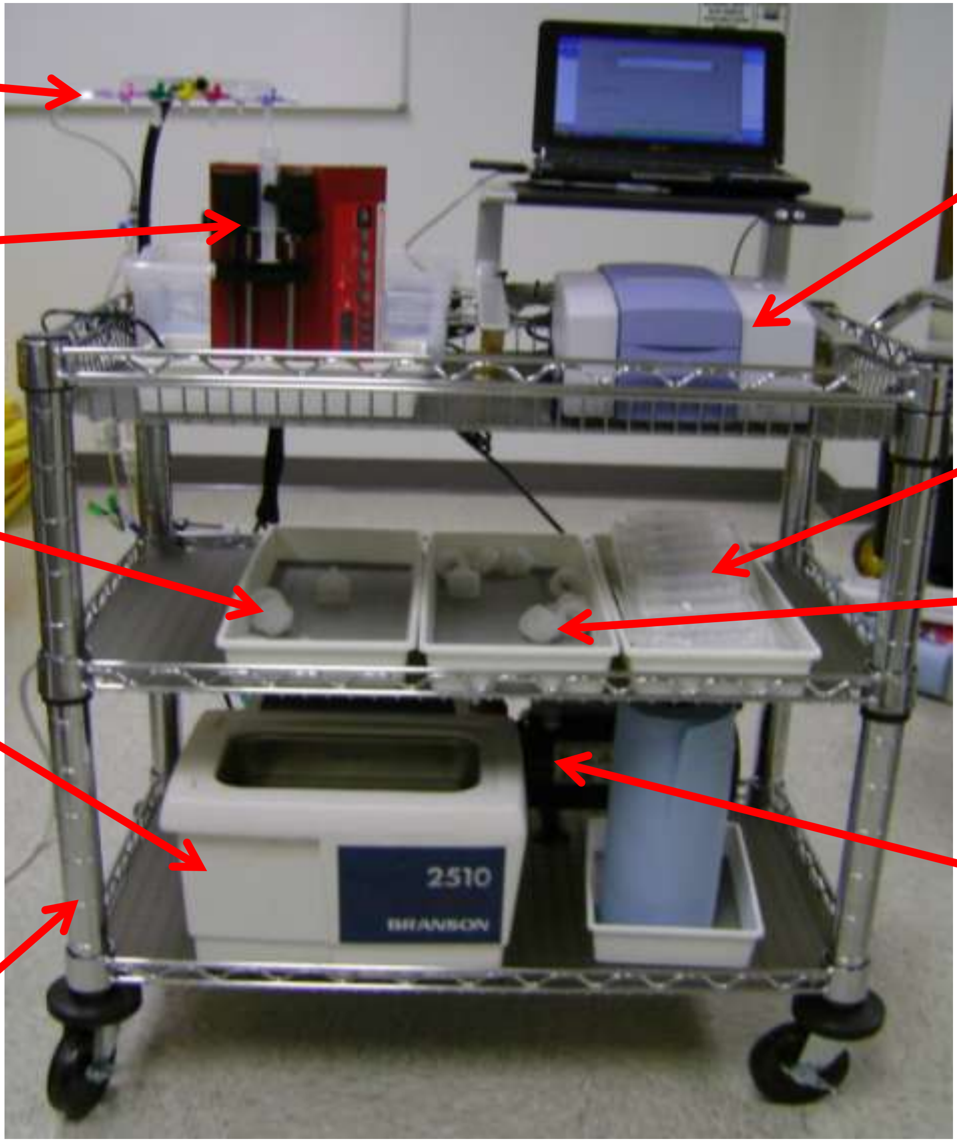
Process Spectrum with
Software…
(< 1 minute)

…To Determine Oil and Grease Content = 101 mg/L

OSS Extractor Part #1018SPE



ASTM D7575 Oil and Grease System



Drying Manifold

Syringe Pump

OSS CSDs

Ultrasonic Bath

Cart
36x32x24

FTIR

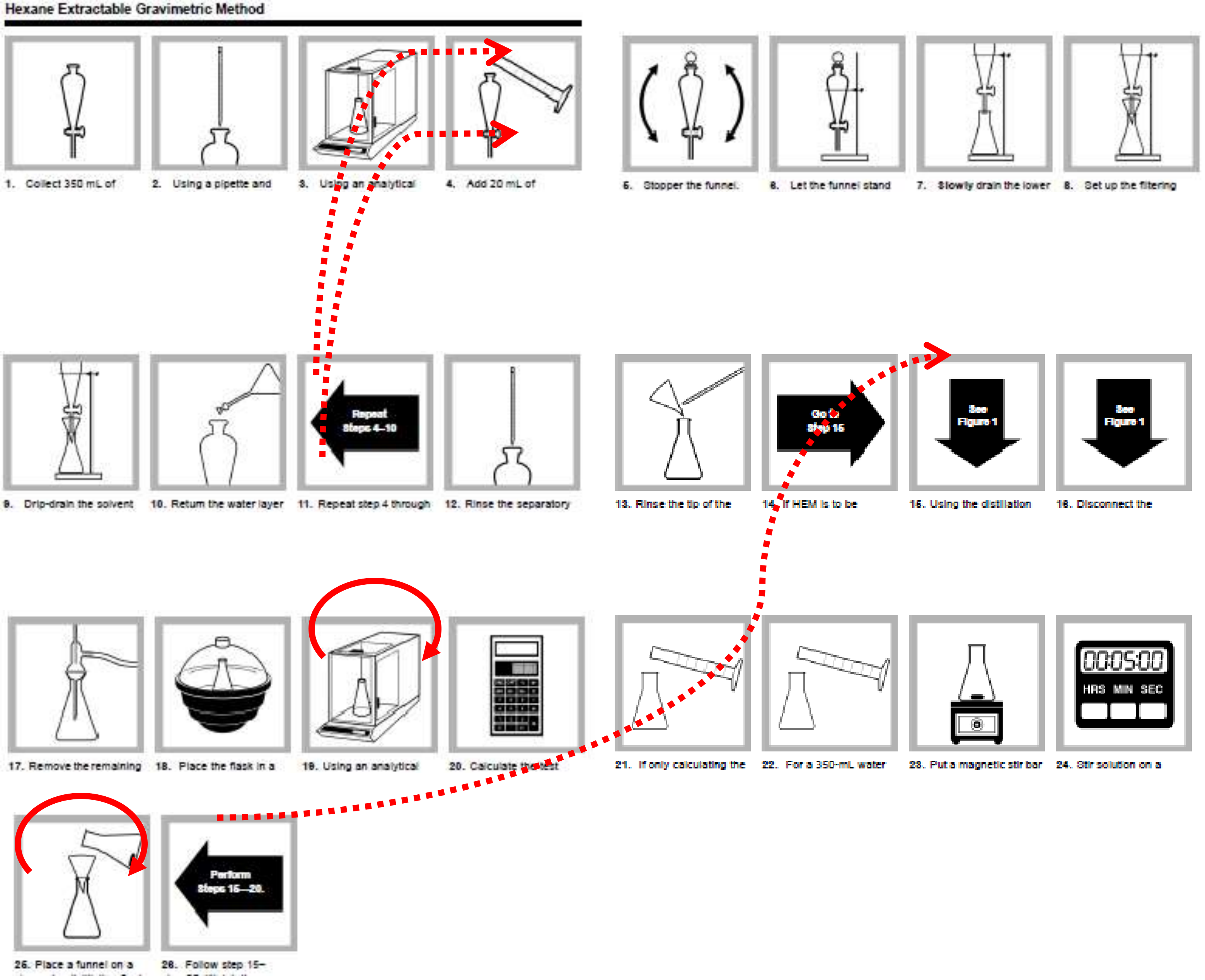
10cc Syringes

OSS
Extractors

Small Air
Compressor
(in back)

ASTM D7575 vs. EPA 1664 METHOD COMPARISON

EPA 1664 METHOD STEPS



Hexane Extractable Gravimetric Method

1. Collect 350 mL of
2. Using a pipette and
3. Using an analytical
4. Add 20 mL of
5. Stopper the funnel.
6. Let the funnel stand
7. Slowly drain the lower
8. Set up the filtering
9. Drip-drain the solvent
10. Return the water layer
11. Repeat step 4 through
12. Rinse the separatory
13. Rinse the tip of the
14. If HEM is to be
15. Using the distillation
16. Disconnect the
17. Remove the remaining
18. Place the flask in a
19. Using an analytical
20. Calculate the test
21. If only calculating the
22. For a 350-mL water
23. Put a magnetic stir bar
24. Stir solution on a
25. Place a funnel on a
26. Follow step 15–

EPA 1664 EQUIPMENT LIST (page 1 of 3)

ASTM D7575 EQUIPMENT

6.1 Sampling equipment.

6.1.1 Sample collection bottles—Glass, approximately 1-L,
NOTE: In those instances necessitating collection of a sample l
or suspected to contain >500 mg/L of HEM (Section 8.1.2), a s
sample container may be used.

6.1.2 Cleaning.

6.1.2.1 Bottles—Detergent water wash, tap water rinse, cap
bake at 200–250EC for 1 h minimum prior to use. Solvent rinse
in place of baking.

6.1.2.2 Liners for screw caps—Detergent water wash, tap w
and bake at 110–200EC for 1 h minimum prior to use.

Method 1664
4 February 1999

6.1.3 Bottles and liners must be lot-certified to be free of a
blanks according to this method (per Section 9.4). If blanks fro
without cleaning or with fewer cleaning steps than required abo
materials, the bottle and liner cleaning steps that do not elimina
be omitted.

6.2 Equipment for glassware cleaning.

6.2.1 Laboratory sink with overhead fume hood.

6.2.2 Oven—Capable of maintaining a temperature within ±

6.3 Equipment for calibration.

6.3.1 Analytical Balance—Capable of weighing 0.1 mg.

6.3.2 Volumetric flask—Glass, 100-mL.

6.3.3 Vials—Assorted sizes, with PTFE-lined screw caps.

6.3.4 Volumetric pipette—Glass, 5-mL.

6.2 Equipment for sample extraction.

6.4.1 Balance (optional)—Top loading, capable of weighing 500–2000 g within ± 1%

6.4.2 Glass stirring rod.

6.4.3 Separatory funnel—Glass, 2000-mL, with PTFE stopcock.

6.4.4 Funnel—Large, glass, for pouring sample into separatory funnel.

6.4.5 Centrifuge (optional)—EXPLOSION PROOF, capable of spinning at least four 100-mL
glass centrifuge tubes at 2400 rpm minimum.

6.4.6 Centrifuge tubes (optional)—100-mL glass.

6.5 Equipment for removal of water, sodium sulfate, and silica gel fines.

6.5.1 Funnel—Analytical, glass.

6.5.2 Filter paper—Whatman No. 40 (or equivalent), to fit funnel.

6.6 Equipment for solvent distillation.

6.6.1 Water bath or Steam bath—EXPLOSION-PROOF, capable of maintaining a temperature
of at least 85EC.

6.6.2 Flask—Boiling, 125-mL (Corning No. 4100 or equivalent).

EPA 1664 EQUIPMENT LIST (page 2 of 3)

6.6.3 Distilling head—Claisen (VWR Scientific No. 26339-005, or equivalent), includes Claisen-type connecting tube and condenser.

6.6.4 Distilling adaptor (attached to the distilling head and to the distillate collection flask for recovery of solvent).

6.6.5 Distillate collection flask (attached to the distilling adaptor for collection of the distilled solvent).

6.6.6 Ice bath or recirculating chiller (to aid in the condensation and collection of the distilled solvent).

6.6.7 Vacuum—Vacuum pump or other source of vacuum.

6.6.8 Tongs, for handling the boiling flask (Baxter Scientific Products No. T5007-2, or equivalent).

6.6.9 Desiccator—Cabinet- or jar-type, capable of keeping the boiling flask (Section 6.6.2) dry during cooling.

6.6.10 Hood-EXPLOSION-PROOF, capable of accommodating the equipment used for solvent distillation (Section 6.6.1-6.6.5).

6.7 Equipment for removal of adsorbable materials.

Method 1664

February 1999 5

6.7.1 Magnetic stirrer.

6.7.2 PTFE-coated magnetic stirring bars.

6.7.3 Graduated cylinder—500-mL, capable of measuring ± 5 mL.

6.7.4 Pipettes—Assorted sizes, calibrated to within ± 0.5 percent.

7.0 Reagents and Standards

7.1 Reagent water—Water in which HEM is not detected at or above the minimum level (ML) of this method. Bottled distilled water or water prepared by passage of tap water through activated carbon have been shown to be acceptable sources of reagent water.

7.2 Hydrochloric acid or sulfuric acid—ACS. Mix equal volumes of concentrated HCl and reagent water or 1 part H_2SO_4 and 3 parts reagent water to produce an approximately 6N solution.

7.3 n-Hexane—85% minimum purity, 99.0% min. saturated C6 isomers, residue less than 1 mg/L.

7.4 Acetone—ACS, residue less than 1 mg/L.

7.5 Sodium sulfate—ACS, granular anhydrous. Dry at 200-250 EC for 24 h minimum and store in a tightly sealed container until use.

NOTE: Powdered sodium sulfate should not be used because traces of water may cause it to solidify.

7.6 Boiling chips—Silicon carbide or fluoropolymer.

7.7 Silica gel—Anhydrous, 75 - 150 micrometers, Davisil Grade 923 (Supelco 21447-7A, or equivalent). Dry at 200–250EC for 24 h minimum and store in a desiccator or tightly sealed container. Determine the n-hexane soluble material content of the silica gel by extracting 30 g of silica gel with n-hexane and distilling the n-hexane to dryness. The silica gel must contain less than 5 mg of n-hexane soluble material per 30 g (< 0.17 mg/g).

EPA 1664 EQUIPMENT LIST (page 3 of 3)

7.8 Hexadecane—98% minimum purity.
7.9 Stearic acid—98% minimum purity.
7.10 Hexadecane/stearic acid (1:1) spiking solution—Prepare in acetone at a concentration of 2
mg/mL each.
7.10.1 Place 200 ± 2 mg stearic acid and 200 ± 2 mg hexadecane in a 100-mL volumetric
flask and fill to the mark with acetone.
NOTE: The solution may require warming for complete dissolution of
stearic acid.
7.10.2 After the hexadecane and stearic acid have dissolved, transfer the solution to a
100–150 mL vial with fluoropolymer-lined cap. Mark the solution level on the vial
and store in the dark at room temperature.
7.10.3 Immediately prior to use, verify the level on the vial and bring to volume with
acetone, if required. Warm to redissolve all visible precipitate.
NOTE: If there is doubt of the concentration, remove 10.0 ± 0.1 mL with
a volumetric pipet, place in a tared weighing pan, and evaporate to
dryness in a fume hood. The weight must be 40 ± 1 mg. If not, prepare
a fresh solution (Section 7.10.1).
Method 1664
6 February 1999
7.11 Precision and recovery (PAR) standard—Using a pipet, spike 10.0 ± 0.1 mL of the
hexadecane/stearic acid spiking solution (Section 7.10) into 950–1050 mL of reagent water
to produce concentrations of approximately 20 mg/L each of hexadecane and stearic acid.
The PAR standard is used for the determination of initial (Section 9.2.2) and ongoing
(Section 9.6) precision and recovery.
7.12 The spiking solutions should be checked frequently for signs of degradation or evaporation
using the test noted in Section 7.10.3, and must be replaced after six months, or sooner if
degradation has occurred.

CWA Effluent Report

Search Criteria

Permit ID: NJ0028878
Discharge points: 001
Parameters: Hydrocarbons, in H2O, IR, CC14 extractible chromatograph
Monitoring locations:
Outfall types:
Sampling periods:
Charts selection: Charts with violations

Permit ID: MS0034711
Discharge points:
Parameters: Oil & grease (freon extr.-IR meth) tot, RC
Monitoring locations: Effluents only
Outfall types: Effluents only
Sampling periods:
Charts selection: Charts with violations

Permit ID: NJ0064921
Discharge points:
Parameters: Hydrocarbons, in H2O, IR, CC14 extractible chromatograph
Monitoring locations: Effluents only
Outfall types: Effluents only
Sampling periods:
Charts selection: Charts with violations

GREEN ASPECT OF ASTM D7575
(Toxic Issues with EPA 1664)

ASTM D7575 – Green!

(this matrix developed with Richard Engler USEPA Green Chemistry Office)

EPA's 12 Principles of Green Chemistry	OSS Extractor	Note
1) Prevention	**YES**	No waste to treat/clean
2) Atom economy	N/A	
3) Less hazardous chem. synthesis	N/A	
4) Designing safer chemicals	**YES**	Non-toxic extractor design
5) Safer solvents and auxiliaries	**YES**	**Solventless!!**
6) Design for energy efficiency	**YES**	Minimal energy use, no fume hoods, no evaporators, no large quantities of hazmat materials to transport
7) Use renewable feedstock	**YES**	Uses small amount of plastic and metal material that can be reused/recycled
8) Reduce derivatives	N/A	
9) Catalysis	N/A	
10) Design for degradation	N/A	Uses small amounts of plastic and metal materials that may be reused / recycled.
11) Analyze in real time to prevent pollution	**YES**	Analysis can be performed on-site and in real-time, portable technology
12) Inherently safer chemistry for accident prevention	**YES**	No solvents or hazardous materials, safe analysis process minimal potential of chemical accident

EPA
United States Environmental Protection Agency

The Presidential Green Chemistry Challenge Awards Program:

Summary of 2010 Award Entries and Recipients

GREEN CHEMISTRY CHALLENGE

An electronic version of this document is available at:
http://www.epa.gov/greenchemistry

Modular Genetics, Inc.

An Acyl Amino Acid Surfactant Produced by Sustainable Chemistry

Surfactants are generally composed of both hydrophobic and hydrophilic regions. Their amphiphilic nature enables them to reduce either the surface tension of a liquid or the interfacial tension between two liquids. These properties enable wide commercial use of surfactants as foaming agents, emulsifiers, and dispersants. Globally each year, petroleum is the feedstock for about 7.4 million metric tons of surfactants; seed oils such as palm or coconut oil are feedstocks for about 3.3 million metric tons. Manufacturing surfactants from petroleum releases approximately 31.6 billion kilograms of carbon dioxide (CO_2) into the atmosphere annually. Manufacturing them from seed oil releases less CO_2, but rainforest preservation limits the available amounts of palm and coconut oils. Sustainably manufactured surfactants have the potential to reduce rainforest destruction and eliminate annual emissions of atmospheric CO_2 equivalent to combusting 3.6 billion gallons of gasoline.

One of the most powerful known biosurfactants is surfactin, a lipopeptide produced by some strains of the soil bacterium *Bacillus subtilis*. Surfactin has only limited use in consumer products, however, because it is not water-soluble. Using genetic techniques, Modular Genetics engineered a *B. subtilis* strain and its peptide synthetase enzyme. The engineered bacterium now produces a novel acyl amino acid surfactant by fermentation of cellulosic carbohydrates such as soybean hulls, an abundant agricultural waste. The new surfactant is a fatty acid-glutamate (FA-Glu) that is very similar to myristoyl glutamate, a commercial surfactant widely used in consumer product formulations. FA-Glu has higher water solubility and a lower critical micelle concentration (CMC) than myristoyl glutamate. Like surfactin, FA-Glu is secreted into the fermentation broth, enabling partial purification by foam fractionation. This novel approach should enable the sustainable manufacture of many new useful surfactants.

During 2009, Modular Genetics distributed samples to potential customers and filed a patent application for its technology.

Orono Spectral Solutions

Device and Method for Analyzing Oil and Grease in Wastewaters without Solvent

Oil and grease are grouped together as one of five conventional pollutants covered by the 1974 Clean Water Act. They rank second only to pH as the most-enforced-against pollutant. All National Pollution Discharge Elimination Systems (NPDES) permits, all pretreatment permits, and all Industrial Effluent Guidelines require measurements of oil and grease. Millions of analyses for oil and grease are done each year in the United States alone.

As a result of the Montreal Protocol in 1989, EPA moved from a Freon extraction method (EPA 413) to an *n*-hexane extraction mass-based determination method (EPA 1664 in 1995; EPA 1664a in 1999). This created several new problems: (1) *n*-hexane methods require personal exposure, handling, and transportation of a hazardous, flammable liquid; (2) *n*-hexane is a known neurotoxin; (3) each analysis takes longer; and (4) millions of liters per year of *n*-hexane waste now require disposal. Thus, the current methodology is inconsistent with the intent of the Clean Air Act and Clean Water Act, both of which consider *n*-hexane a hazardous pollutant.

The "solid phase infrared attenuable extractor" technology developed by Orono Spectral Solutions (OSS) solves the problems above by eliminating solvents from oil and grease analysis. It also provides more economical, accurate analyses. OSS's extractor unit is small, economical, robust, and disposable (or partially recyclable). It contains no toxic substances. It includes a polymeric membrane that captures and concentrates oil and grease from water, a metal-membrane support, and a polypropylene housing designed for pressurized water

22

UNIVERSITY OF CALIFORNIA, BERKELEY

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Berkeley, CA 94720-7360

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U.S. 510-642-5703
International: 011-1-510-642-5703

U.S. Environmental Protection Agency;
EPA Docket Center (EPA/DC) Water Docket
MC 28221T
1200 Pennsylvania Avenue, NW
Washington, DC 20460

Docket ID No. EPA-HQ-OW-2010-0192

The letter presents three key points:

1) n-Hexane is a well-documented occupational health hazard;

2) Health effects from exposure to n-hexane can occur at exposure levels several orders of magnitude below the federal Occupational Safety and Health Administration's Permissible Exposure Limit of 1,800 mg/m^3 (500 ppm);

3) Reducing the commercial circulation of n-hexane by allowing methods that use solvent-free technologies would help mitigate worker exposures to n-hexane throughout its life cycle; this approach is consistent with EPA's commitment to the principles of green chemistry.

United States
Environmental Protection
Agency

Office Of Water
(EN-336)

EPA 812-B-92-001
NTIS No. PB92-173-236
June 1992

EPA

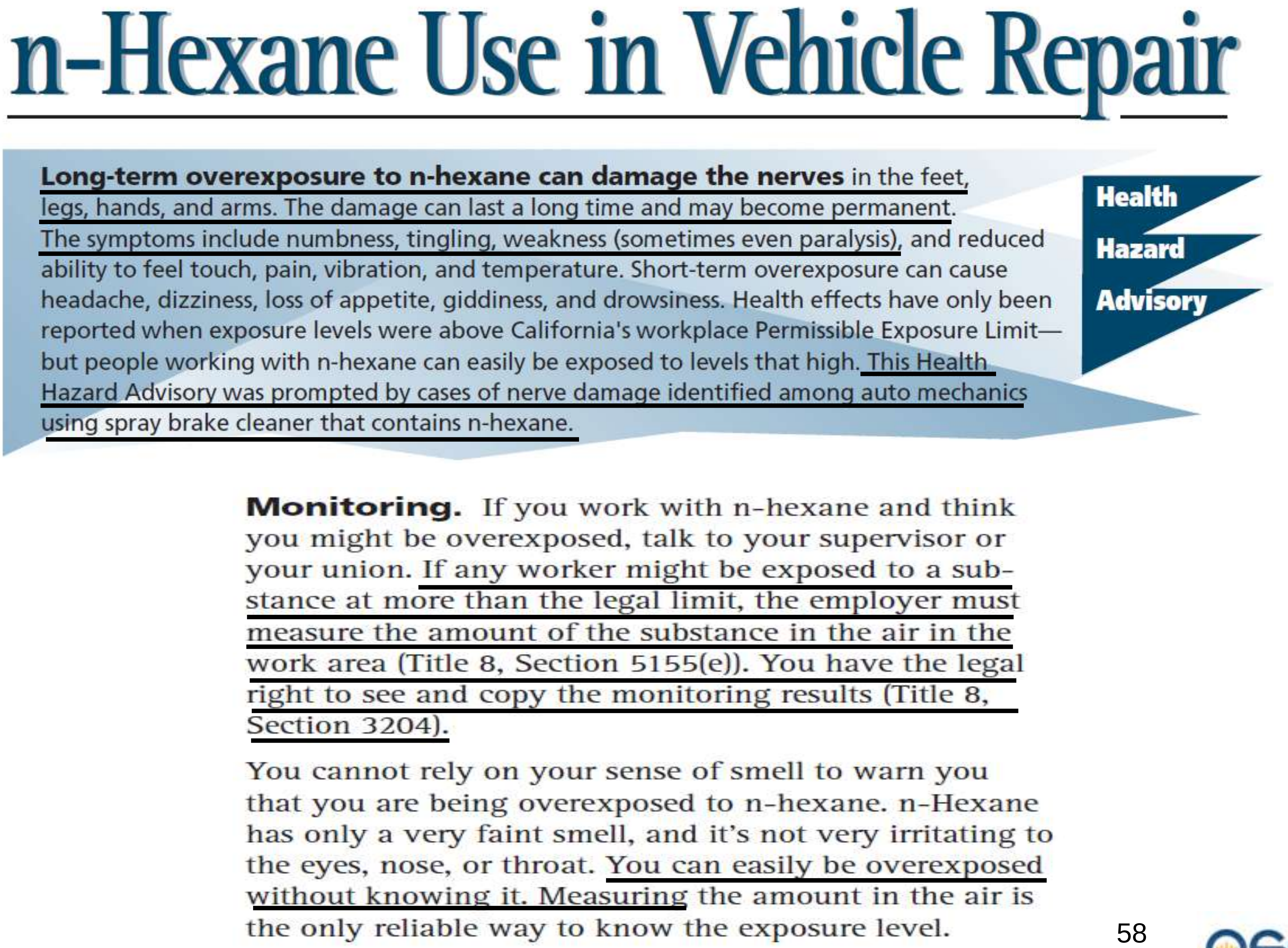
Guidance To Protect POTW Workers From Toxic And Reactive Gases And Vapors

Hexane Mentioned 38 times!!!

Cases of direct health issues
Cases of explosions

Air-reactive substances - These substances may cause fires or explosions, and may generate toxic gases, or vapors.

Flammable air-reactives - include hydrocarbon solvents (such as **hexane**, toluene, naphtha) and fuels (such as gasoline).



n-Hexane Use in Vehicle Repair

Health
Hazard
Advisory

Long-term overexposure to n-hexane can damage the nerves in the feet, legs, hands, and arms. The damage can last a long time and may become permanent. The symptoms include numbness, tingling, weakness (sometimes even paralysis), and reduced ability to feel touch, pain, vibration, and temperature. Short-term overexposure can cause headache, dizziness, loss of appetite, giddiness, and drowsiness. Health effects have only been reported when exposure levels were above California's workplace Permissible Exposure Limit—but people working with n-hexane can easily be exposed to levels that high. This Health Hazard Advisory was prompted by cases of nerve damage identified among auto mechanics using spray brake cleaner that contains n-hexane.

Monitoring. If you work with n-hexane and think you might be overexposed, talk to your supervisor or your union. If any worker might be exposed to a substance at more than the legal limit, the employer must measure the amount of the substance in the air in the work area (Title 8, Section 5155(e)). You have the legal right to see and copy the monitoring results (Title 8, Section 3204).

You cannot rely on your sense of smell to warn you that you are being overexposed to n-hexane. n-Hexane has only a very faint smell, and it's not very irritating to the eyes, nose, or throat. You can easily be overexposed without knowing it. Measuring the amount in the air is the only reliable way to know the exposure level.

OIL IN WATER TECHNOLOGIES MATRIX

ORONO SPECTRAL SOLUTIONS, INC.
A Full Spectrum Innovator
www.ossmaine.com

OIL IN WATER TECHNOLOGY MATRIX

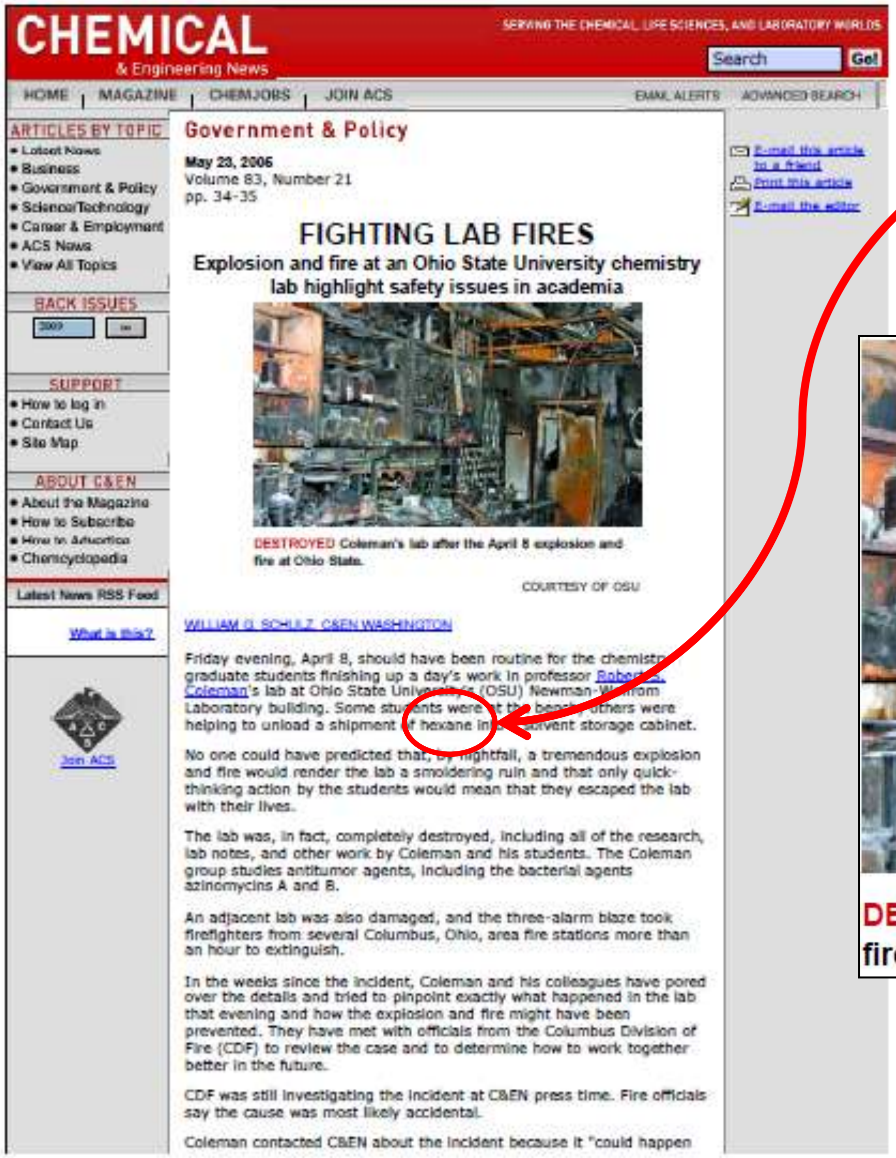
Attribute Technology	Solventless	Quick, Easy Analysis	Direct Oil Measurement	CFC Free	Compatible with Current & Previous Methods	Minimal Operating Logistics	Low Total Cost of Ownership
OSS	✓	✓	✓	✓	✓	✓	✓
Historical IR	✗	✗	✓	✗	✗	✗	✗
Gravimetric	✗	✗	✓	✓	✓	✗	✗
GC - FID	✗	✗	✓	✓	✓	✗	✗
Fluorescence	✓	✓	✗	✓	✗	✓	?
Light Scattering	✓	✗	✗	✓	✗	✓	?

OSS TECHNOLOGY FEATURES

NEXT-GENERATION OIL IN WATER ANALYSIS

- ☑ Environmentally Friendly
- ☑ Solventless (No CFC!)
- ☑ Economical, Accurate DIRECT Measurement of ALL Hydrocarbons
- ☑ Correlates to EPA, ASTM, and OSPAR Methods
- ☑ **Infrared Based**
- ☑ Patent Pending

HEXANE EXPLODES OHIO STATE UNIVERSITY LABORATORY



Government & Policy

May 23, 2005
Volume 83, Number 21
pp. 34-35

FIGHTING LAB FIRES

Explosion and fire at an Ohio State University chemistry lab highlight safety issues in academia

DESTROYED Coleman's lab after the April 8 explosion and fire at Ohio State.

COURTESY OF OSU

WILLIAM G. SCHULZ, C&EN WASHINGTON

Friday evening, April 8, should have been routine for the chemistry graduate students finishing up a day's work in professor Robert S. Coleman's lab at Ohio State University's (OSU) Newman-Wolfrom Laboratory building. Some students were at the bench; others were helping to unload a shipment of hexane into a solvent storage cabinet.

No one could have predicted that, by nightfall, a tremendous explosion and fire would render the lab a smoldering ruin and that only quick-thinking action by the students would mean that they escaped the lab with their lives.

The lab was, in fact, completely destroyed, including all of the research, lab notes, and other work by Coleman and his students. The Coleman group studies antitumor agents, including the bacterial agents azinomycins A and B.

An adjacent lab was also damaged, and the three-alarm blaze took firefighters from several Columbus, Ohio, area fire stations more than an hour to extinguish.

In the weeks since the incident, Coleman and his colleagues have pored over the details and tried to pinpoint exactly what happened in the lab that evening and how the explosion and fire might have been prevented. They have met with officials from the Columbus Division of Fire (CDF) to review the case and to determine how to work together better in the future.

CDF was still investigating the incident at C&EN press time. Fire officials say the cause was most likely accidental.

Coleman contacted C&EN about the incident because it "could happen

http://pubs.acs.org/cen/news/83/i21/8321ohio.html[12/18/2010 10:37:37 PM]

DESTROYED Coleman's lab after the April 8 explosion and fire at Ohio State.

Summary

OSS METHOD:

NO SOLVENTS

NO TOXIC SUBSTANCES

DISPOSABLE

FAST, EASY PROCESS

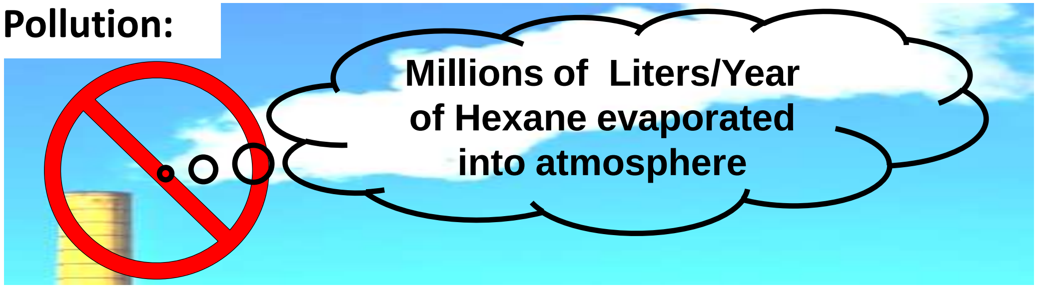
MEETS OR EXCEEDS EPA 1664 PERFORMANCE

CURRENT EPA 1664 METHOD:

Hexane Issues:

- Danger of serious health damage
- Irritating to skin, eyes, lungs
- Risk of impaired fertility
- Toxic to aquatic organisms

Pollution:



Millions of Liters/Year of Hexane evaporated into atmosphere

Hazardous Materials Transport / Disposal:

SUPPORT FOR ASTM D7575 INCLUSION IN METHOD UPDATE RULE

Bill Telliard Points of Note re. ASTM D7575 Development

Here are some points for you all to keep in mind:

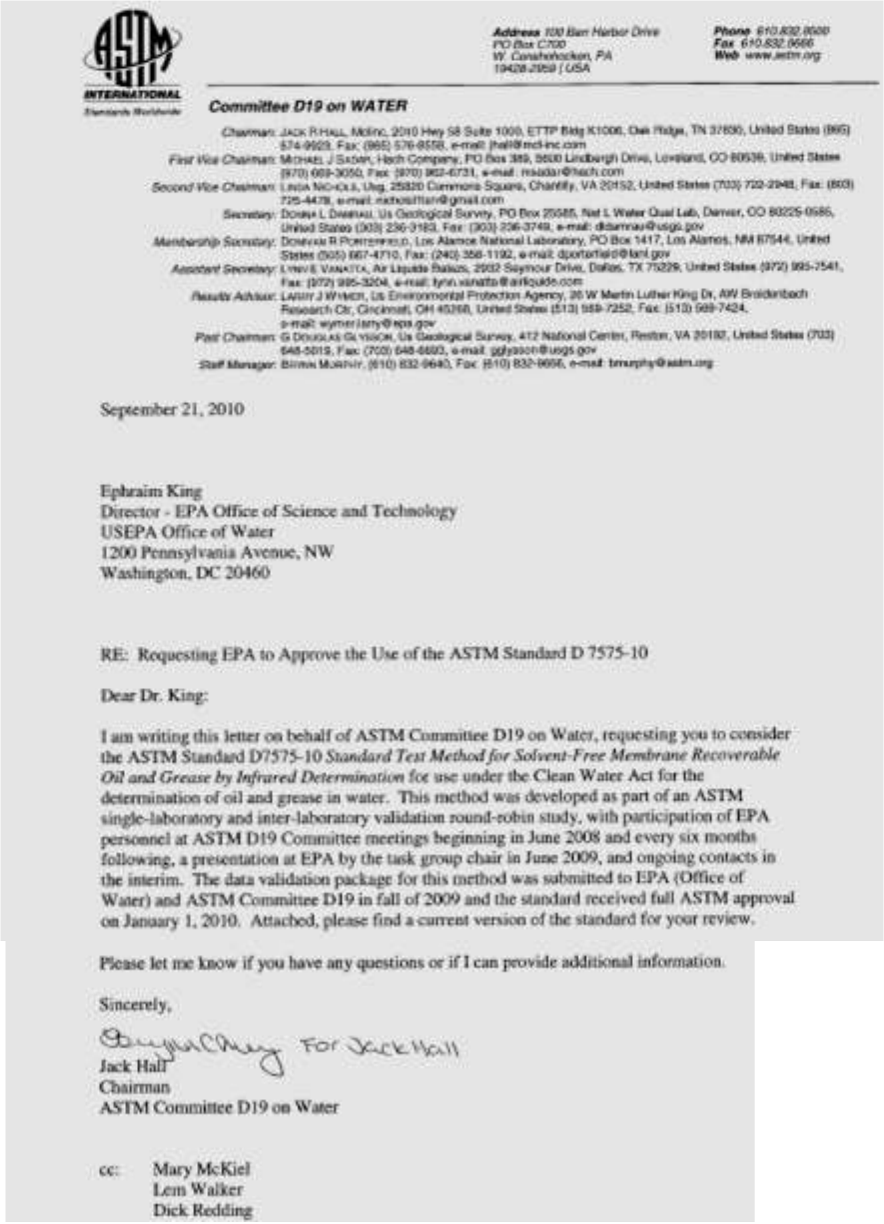
1) A comparison study between 1664 and D7575 was carried out in accordance with the procedures in EPA's Phase II Freon Replacement Study.

2) On page 5 of the EPA Freon Replacement Study, the Agency states that infrared techniques/methods would be evaluated in the Phase III Replacement Study. Obviously, infrared was under consideration by the Agency.

3) For D7575, a side-by-side comparison was carried out, in triplicate, by a single, independent laboratory. And this was also the case with the EPA Phase II Study. Again, in D7575, thirty samples are covered in the ASTM Report. Also three (or four) additional samples were analyzed, and the results sent to the Agency--separately.

4) The EPA Phase II Freon Replacement Study analyzed 34 samples. The ASTM D7575 Study analyzed 33 samples. We did not sample a more diverse collection of industries because we did not have 308 Authority—the device EPA employed for its collection efforts. Note that the volunteer industries that we employed for the D7575 ASTM Study were approved by EPA (Dick Reding).

ASTM Formal Request to EPA to Include D7575 in Rule



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Results Advisor: LARRY J WYMER, Us Environmental Protection Agency, 26 W Martin Luther King Dr, AW Breidenbach Research Ctr, Cincinnati, OH 45268, United States (513) 569-7252, Fax: (513) 569-7424, e-mail: wymer.larry@epa.gov
Past Chairman: G DOUGLAS GLYSSON, Us Geological Survey, 412 National Center, Reston, VA 20192, United States (703) 648-5019, Fax: (703) 648-6693, e-mail: gglysson@usgs.gov
Staff Manager: BRYNN MURPHY, (610) 832-9640, Fax: (610) 832-9666, e-mail: bmurphy@astm.org

September 21, 2010

Ephraim King
Director - EPA Office of Science and Technology
USEPA Office of Water
1200 Pennsylvania Avenue, NW
Washington, DC 20460

RE: Requesting EPA to Approve the Use of the ASTM Standard D 7575-10

Dear Dr. King:

I am writing this letter on behalf of ASTM Committee D19 on Water, requesting you to consider the ASTM Standard D7575-10 *Standard Test Method for Solvent-Free Membrane Recoverable Oil and Grease by Infrared Determination* for use under the Clean Water Act for the determination of oil and grease in water. This method was developed as part of an ASTM single-laboratory and inter-laboratory validation round-robin study, with participation of EPA personnel at ASTM D19 Committee meetings beginning in June 2008 and every six months following, a presentation at EPA by the task group chair in June 2009, and ongoing contacts in the interim. The data validation package for this method was submitted to EPA (Office of Water) and ASTM Committee D19 in fall of 2009 and the standard received full ASTM approval on January 1, 2010. Attached, please find a current version of the standard for your review.

Please let me know if you have any questions or if I can provide additional information.

Sincerely,

For Jack Hall
Jack Hall
Chairman
ASTM Committee D19 on Water

cc: Mary McKiel
Lem Walker
Dick Redding

MULTILAB STUDY – USER SURVEY EXAMPLES:

“I was thoroughly impressed with the ease of use and the speed of this method. Even with minimal training, I felt comfortable and confident when asked to use the method without assistance. It only took a couple hours to process an MDL and several samples. With our current system, this could take as much as a full day to run. As the Health and Safety Officer, I was very glad to see that this is a solvent-free method. Any way to reduce hazardous materials in the lab is great. It’s safer and helps save money. Another of my responsibilities is disposal of hazardous waste. We currently collect 2 X 1 liter ambers for Oil and Grease Analysis. It seems more than likely, that with this method, as long as it doesn’t affect reporting limits; a smaller volume could be collected. “

“One large cost- and health-saving advantage of this method is that it does not use any solvent. It can also be a space-saving advantage, since it does not need to be performed in a fume hood, allowing other analyses to be performed in the hood while the Oil and Grease analysis is also being performed. “

CURRENT METHOD UPDATE RULE (EPA-HQ-OW-0192)

9 INDEPENDENT ASTM D7575 PUBLIC COMMENT EXAMPLES

*** Note these are comments even though ASTM D7575 was NOT PROPOSED

1) Comment attachment submitted by Andrea Rex, Massachusetts Water Resources Authority
Comments Due Dec 22, 2010 11:59 PM ET Public Submission Massachusetts Water Resources Authority EPA-HQ-OW-2010-0192-0091.1 11/22/2010
“For oil and grease, this section explicitly DOESN’T propose for approval ASTM method D7575-10, which is based on solid phase extraction using a membrane and quantitation by infrared absorption of the membrane. We had occasion to try this method out with the method developer (OSS) and **found it to be simple yet elegant. It worked well on method detection limit and precision and accuracy samples**,..”

2) Comment attachment submitted by Willie R. Taylor, Director, Office of Environmental Policy and Compliance, U.S. Department of the Interior
Comments Due Dec 22, 2010 11:59 PM ET Public Submission EPA-HQ-OW-2010-0192-0108.1 12/01/2010
Proposed Revisions to Method Modification Provisions at 40 CFR 136 “We are in agreement that regulatory authorities should allow analysts the flexibility to modify CW A methods without prior approval provided the user has documented **equivalent or better performance** of the method in that particular matrix type. In instrumental analyses. analysts may need to modify approved methods in order to combat matrix interferences and these modifications should not require extensive review. The Department agrees that **acceptable reasons** for these modifications should include **lower detection limits, improved precision, less spectral interference, lower laboratory cost and the reduction of laboratory waste**. “

3) Comment submitted by Andre Brousseau, President, Maine Wastewater Control Association (MWWCA)
Comments Due Dec 22, 2010 11:59 PM ET Public Submission Maine Wastewater Control Association (MWWCA) EPA-HQ-OW-2010-0192-0109
“*Method 1664 for Oil& Grease: Hexane*
In the Federal Register of September 23, 2010, EPA seeks to approve new technologies for other test parameters as part of its changes to 40 CFR 136, yet **falls short of being able to accept new methods that include new technologies for Oil & Grease.** NfWWCA finds that the EPA's reasoning for not approving alternative test methods for Oil & Grease is **contradictory** to the Agency's "Summary" statement that these regulations will "provide increased flexibility to the regulated community and laboratories in their selection of analytical methods (test procedures) for use in Clean Water Act programs." Further, approving the new methods would be more consistent with the EPA's mission and purpose to **"ensure that all Americans are protected from significant risks to human health and the environment where they live learn and work. “ EPA has previously approved hexane extractable methods for Oil & Grease, then reversed that decision because hexane was a hazardous solvent and required use of Freon-extractable methods for Oil & Grease. Then EPA reversed that decision** and went back to hexane, because Freon was considered more harmful to human health and the environment than hexane. However, **hexane remains a dangerous solvent.** n-Hexane has a flashpoint of -22 degrees Celsius, so most n-hexane waste is considered a RCRA hazardous waste by characteristic. According to the u.s. Department of Labor, Occupational Safety and Health Administration (OSHA) Guideline for n-Hexane (2008) discussion on toxicology to animals, "n-Hexane is a neurotoxin, a narcotic, and an irritant of the eyes, skin, and mucous membranes [Hathaway et al. 199 I]. n-Hexane also causes (re)productive and embryo toxic effects and is cytotoxic in mammalian and human test systems [NIOSH 1991]." OSHA also found that n-Hexane is toxic to humans, and recommends extensive medical surveillance for persons working with n-Hexane. EPA must consider the goals of the Clean Water Act when approving test procedures for Clean Water Act permittees in 40 CFR 136. **NJWWCA believes that the fats, oils and greases that may be toxic to aquatic organisms, or interfere with sewer collection systems and wastewater treatment, are primarily petroleum, animal or vegetable in origin. These fats, oils and greases are not exclusively "hexane extractable" compounds. In fact, other technologies and methods may be better at measuring these compounds, and may be used to better quantify how much fat, oil or grease is toxic to aquatic life or interferes with wastewater treatment.**
EPA should not specifically and uniquely endorse a solvent specific method for "Oil & Grease." The NJWWCA requests that EPA reverse its decision that only n-hexane extractable Oil & Grease methods are acceptable. We understand that, to further that reversal, EPA must re-write its current definition of oil and grease so that new technologies and test methods can be approved.

CURRENT METHOD UPDATE RULE (EPA-HQ-OW-0192)

9 INDEPENDENT ASTM D7575 PUBLIC COMMENT EXAMPLES (continued)

4) Comment submitted by David N. Speis, Chair, Environmental Laboratory Advisory Board (ELAB)

Comments Due Dec 22, 2010 11:59 PM ET Public Submission Environmental Laboratory Advisory Board (ELAB) EPA-HQ-OW-2010-0192-0120 12/20/2010

“**Comment: Flexibility in the Use of Methods for Different Analytes.**

There is a need for flexibility in adding analytes to EPA-approved methods as long as the supporting quality assurance (QA) and QC based on data quality objectives (DQOs) demonstrates acceptable data quality. As water quality criteria are adopted for new analytes, this type of method flexibility would facilitate more defensible and reliable monitoring of these analytes. Where feasible for a given technology, EPA should allow current EPA approved methods to be used to analyze compounds/analytes in addition to those listed in the methods as long as QA/QC demonstrates acceptable data quality. The development of DQOs for individual programs would facilitate this flexibility and reduce the need for new methods. ELAB recommends that EPA consider providing this flexibility in the final rule.

Comment: Method Validation and Approval Process.

EPA has proposed numerous changes to the methods in the MUR simply by approving different editions of Standard Methods for use. This is significant because the method approval process for Standard Methods is not the same as that for EPA or other standards-setting organizations. Additionally, changes are being made by vendors to methods previously deemed to be equivalent to 40 CFR Part 136 methods (e.g., Lachat Methods for total kjeldahl nitrogen ammonia); the acceptability or approval of these changes or updates is not clear. Based on these observations, **it appears that many changes to the methods approved for use in this rule, given the information provided by EPA, are occurring without a systematic review, validation, and approval process.**

5) Comment submitted by Ellen R Campbell, Nitrate Elmination Co Inc

Comments Due Dec 22, 2010 11:59 PM ET Public Submission Nitrate Elmination Co Inc EPA-HQ-OW-2010-0192-0121 12/20/2010

“General Comment

The methods approval process at the EPA under the Clean Water and Safe Drinking Water Acts is geared toward old, established analytical methods in use before 1960. The EPA seems willing to accept new methods that require high-cost equipment. This mindset is detrimental to innovative companies here in the US. **It is time for the EPA to start considering and approving new methods based on reduction of hazardous or toxic reagents AND on methods that are proven to work,**. Advances in biotechnology and materials science may simplify and reduce costs for many analytes of interest to the EPA. This **How can a new mindset at the EPA toward green and cost-effective methods be established?**”

6) Anonymous public comment

Comments Due Dec 22, 2010 11:59 PM ET Public Submission University of Maine EPA-HQ-OW-2010-0192-0129 12/21/2010

General Comment

As an individual who has performed both the previous Freon method as well as the n-hexane method I have been exposed to these dangerous fumes. I read your documentation for why this specific method ASTM D7575 was not chosen as a viable alternative to EPA 1664 based on the fact that all oil and grease sampling techniques require an organic solvent. This breakthrough in the measurement of oil and grease should be adopted and the rationale that the method doesn't include a solvent is not in the best interest of science or the environment.

CURRENT METHOD UPDATE RULE (EPA-HQ-OW-0192)

9 INDEPENDENT ASTM D7575 PUBLIC COMMENT EXAMPLES (continued)

7) Comment attachment submitted by Paul Wiegand, Vice President, Water Quality, National Council for Air and Stream Improvement, Inc. (NCASI)
Comments Due Dec 22, 2010 11:59 PM ET Public Submission EPA-HQ-OW-2010-0192-0166.1 12/27/2010
“ As detailed in the attachment, **NCASI’s primary concern is that EPA is proposing adoption of many methods despite the absence of validation data, both in general and specifically using real-world matrices**. The absence of appropriate method validation data in the docket is of concern because without such data not only is it impossible to verify the performance of methods proposed as part of this rule, but it also suggests that EPA is proposing methods absent the data to bootstrap even basic quality control (see Comment #2 in the attachment).

Similarly, **EPA proposes addition of several EPA methods (e.g., 1614A, 614.1, 632, 1664 Rev. B, 622.1, and 619) but has not provided any validation data** demonstrating the capabilities of the methods to meet the testing requirements for the NPDES program, nor has it conducted a peer review and made the result available for public comment.”

8) Comment attachment submitted by Joe Boyd, Environmental Express
Comments Due Dec 22, 2010 11:59 PM ET Public Submission EPA-HQ-OW-2010-0192-0181.112/27/2010
“The EPA has decided not to include ASTM method D7575-10 in Table IB mainly due to the fact that this method does not use the solvent n-hexane in the determination of oil & grease. Methods that are currently listed in Table IB (EPA 1664B, SM 5520B-2001, and SM 5520F-2001) do indeed list n-hexane as the solvent to use for extraction. However, all three of these methods reference “oil & grease” as a method defined parameter based on the use of hexane as the extraction solvent. This makes the analytes of interest “hexane extractable material”, or HEM rather than “oil & grease”. While both “HEM” and “oil & grease” can be construed as method defined analytes, HEM is much more specific in that the name itself implies that the analytes of interest must be extractable using hexane. “Oil & grease”, however, is a broader term and does not limit itself to any specific type of extraction. One must understand that no matter what extraction procedure is employed, the analytes of interest will always be a method defined parameter as the term “oil & grease” is truly indefinable. The continued use of the term “oil & grease” in Table IB for Parameter 41, however, leads to some confusion and brings into question the reasoning behind the EPA’s decision to limit the measurement of this parameter to hexane extractable material.

The EPA’s decision to abandon methods 413.1 and 418.1 to eliminate the use of the fluorochlorohydrocarbon Freon® has been widely accepted as a step in the positive direction to reduce the amount of pollution generated by the laboratory. **While n-hexane is an improvement over Freon®, it is still a highly flammable solvent and has the potential to be highly toxic to the user.**

Environmental Express, Inc. does not specifically endorse the product created by Orono Spectral Solutions Inc. (OSS) used in ASTM method D7575-10, however we would ask that the EPA continue to look at products that can make various analyses “greener”. The supporting data provided by OSS does seem to indicate an equivalent option for the determination of the parameter “oil & grease” and eliminates the risks created by the use of n-hexane. While ASTM method D7575-10 does not lead to the analyte of interest as HEM, it does provide an alternative extraction technique to determine “oil & grease”. The EPA has already demonstrated that it is willing to alter the definition of the end analyte of interest for the parameter of “oil & grease” by moving from Freon® to nhexane, creating HEM. The **EPA should keep the spirit of this philosophy alive in the move to greener methodologies and reconsider it’s stance on ASTM D7575-10.**”

CURRENT METHOD UPDATE RULE (EPA-HQ-OW-0192)

9 INDEPENDENT ASTM D7575 PUBLIC COMMENT EXAMPLES (continued)

9) Comment submitted by Peter Halpin, Caltest Analytical Laboratory

Public Submission Caltest Analytical Laboratory EPA-HQ-OW-2010-0192-0187 12/30/2010

“In the spirit of the March 207 Method Update Rule that applied to Clean Water Act analyses and sampling procedures **we are requesting that you include flexibility to reduce costs where feasible and where the changes do not result in a reduction of data quality**. Specifically in allowing alternate extraction procedures, addition of an analyte, and the use of mass spectrometer detectors for all semivolatile analytes where the mass spectrometer is capable of providing sufficiently sensitive detection limits for the purpose of the analysis.

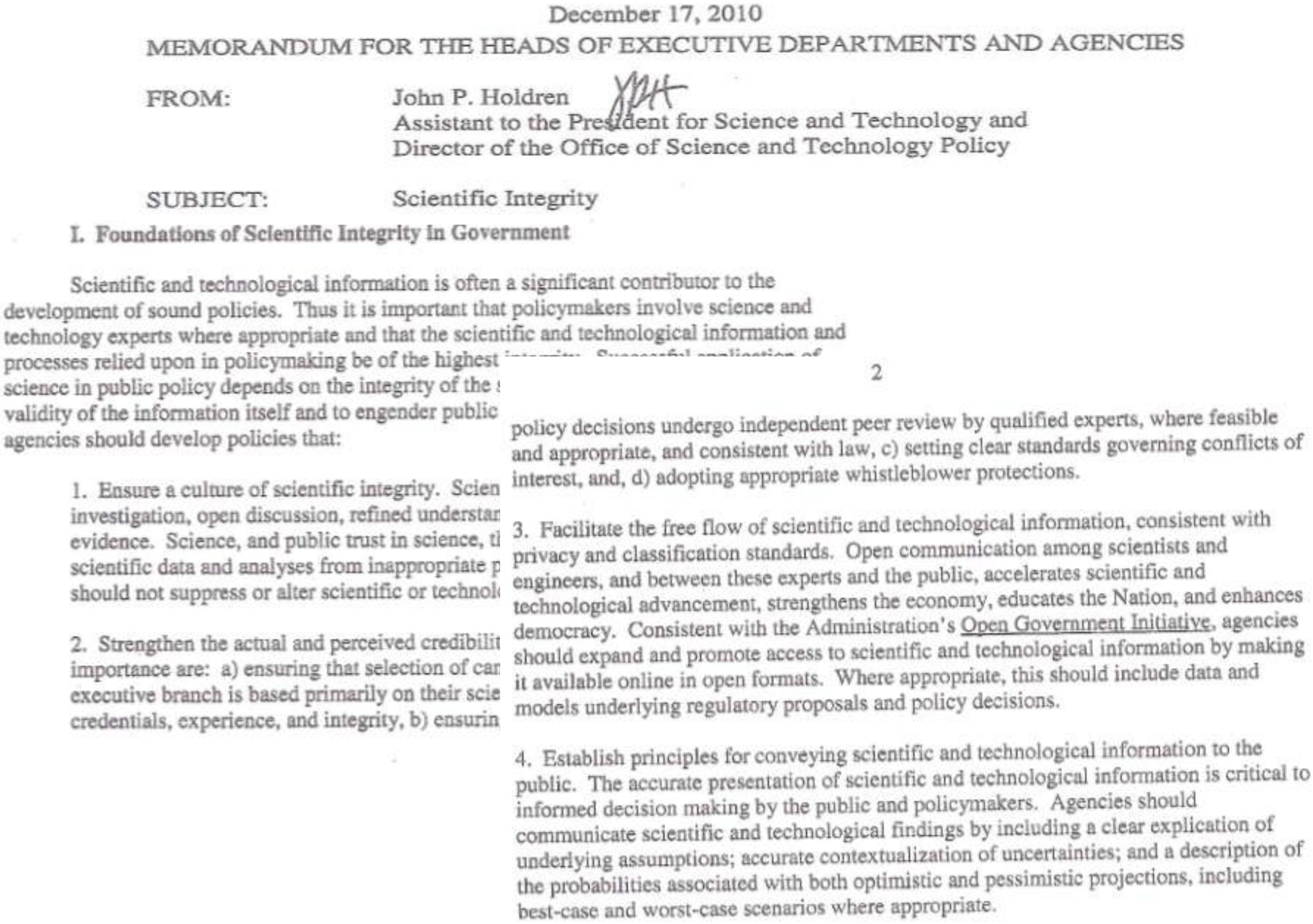
For example, where extractable organics can be demonstrated to be adequately extracted from the target water by solid phase extraction, we advocate allowing the solid phase extraction. This would result in much less use of the toxic methylene chloride solvent, and generate less waste. Some methods explicitly allow solid phase extraction, some are too old to make reference to the technique. **In addition to solid phase extraction, other green chemistry approaches that minimize solvent waste, hazard, and analyst time** include stir bar sorptive extraction, and solid phase microextraction. **When these approaches can be shown to meet the monitoring objective it would be helpful to be able to use them for compliance purposes…”**

Understanding Voluntary Consensus Standards

JoAnne Overman
Standards Services Division
National Institute of Standards and Technology

- National Technology Transfer and Advancement Act (NTTAA) of 1995
  - Directs federal agencies to use “technical standards developed or adopted by the voluntary consensus standards bodies” as a means to carry out policy objectives or activities determined by the agencies or departments

- Office of Management and Budget (OMB) Circular A-119 (1998)
  - Encourages federal agencies to benefit from the expertise of the private sector
  - Promotes agency participation in standards development activities
  - Seeks to reduce reliance on government unique standards
  - Section 6 of the OMB Circular requires head of agencies to provide an explanation of the reason(s) for using government-unique standards in lieu of voluntary consensus standards
  - Annual report prepared by NIST for OMB on government use of standards and participation in standards activities



December 17, 2010

MEMORANDUM FOR THE HEADS OF EXECUTIVE DEPARTMENTS AND AGENCIES

FROM: John P. Holdren
Assistant to the President for Science and Technology and
Director of the Office of Science and Technology Policy

SUBJECT: Scientific Integrity

I. Foundations of Scientific Integrity in Government

Scientific and technological information is often a significant contributor to the development of sound policies. Thus it is important that policymakers involve science and technology experts where appropriate and that the scientific and technological information and
processes relied upon in policymaking be of the highest
science in public policy depends on the integrity of the
validity of the information itself and to engender public
agencies should develop policies that:

1. Ensure a culture of scientific integrity. Scien
investigation, open discussion, refined understan
evidence. Science, and public trust in science, th
scientific data and analyses from inappropriate p
should not suppress or alter scientific or technol

2. Strengthen the actual and perceived credibilit
importance are: a) ensuring that selection of can
executive branch is based primarily on their scie
credentials, experience, and integrity, b) ensurin

policy decisions undergo independent peer review by qualified experts, where feasible and appropriate, and consistent with law, c) setting clear standards governing conflicts of interest, and, d) adopting appropriate whistleblower protections.

3. Facilitate the free flow of scientific and technological information, consistent with privacy and classification standards. Open communication among scientists and engineers, and between these experts and the public, accelerates scientific and technological advancement, strengthens the economy, educates the Nation, and enhances democracy. Consistent with the Administration's Open Government Initiative, agencies should expand and promote access to scientific and technological information by making it available online in open formats. Where appropriate, this should include data and models underlying regulatory proposals and policy decisions.

4. Establish principles for conveying scientific and technological information to the public. The accurate presentation of scientific and technological information is critical to informed decision making by the public and policymakers. Agencies should communicate scientific and technological findings by including a clear explication of underlying assumptions; accurate contextualization of uncertainties; and a description of the probabilities associated with both optimistic and pessimistic projections, including best-case and worst-case scenarios where appropriate.

ASTM D7575 FIELD EXPERIENCE

OSS GULF OIL DISASTER FIELD TESTING
JUNE 2010

Blog at http://www.ossmaine.com/

Tom Schwarz

Dean Smith

GULF FIELD TESTING HIGHLIGHTS:

- Set up equipment and train personnel at Alpha Test Site (Petroleum Laboratories, Lafayette, LA) on ASTM METHOD D7575

- Perform side-by-side comparisons against EPA 1664a

- Perform field sampling around Louisiana coast:
    - Unaffected areas in the west
    - Hard-hit areas in Grand Isle
    - Surface, subsurface, and sediment samples
    - 100+ samples taken and processed

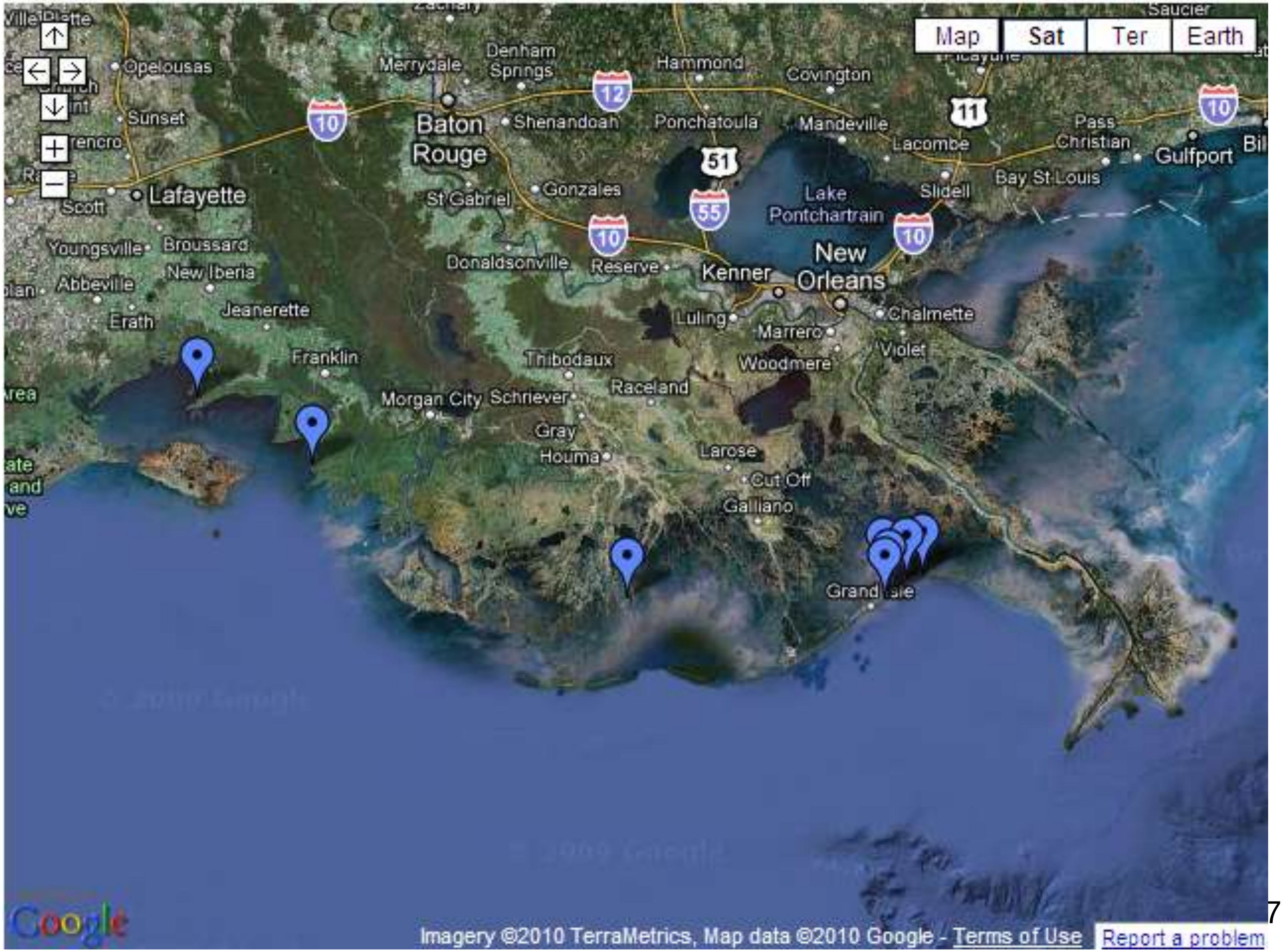
- Perform real-time field analysis of ASTM D757 using only a generator for power.

Alpha Test Site: Petroleum Laboratories Lafayette, LA

Thousands of samples/year

Support of offshore industry

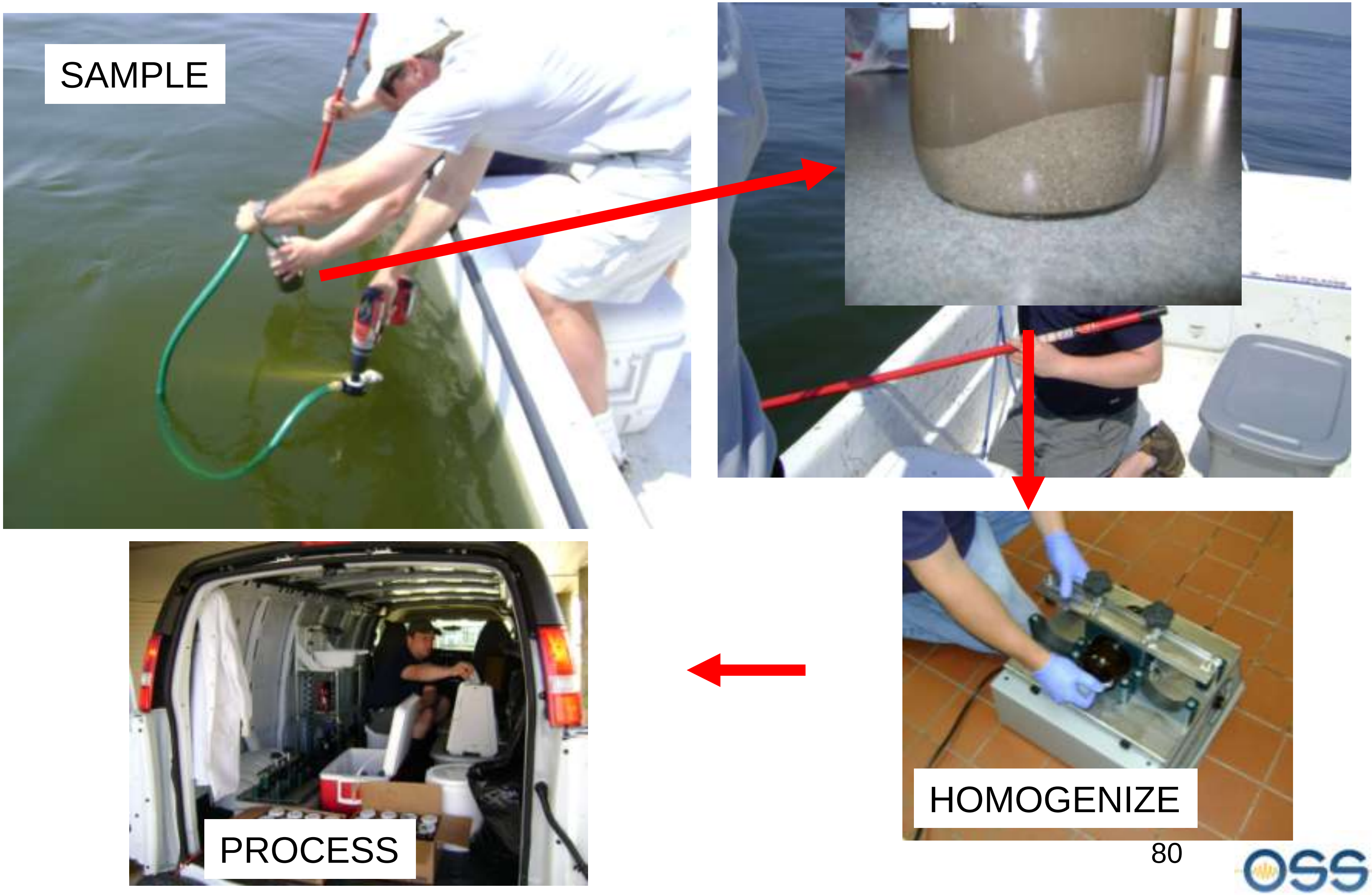
Field Sampling Areas



Field Sampling: Grand Isle (surface grab)

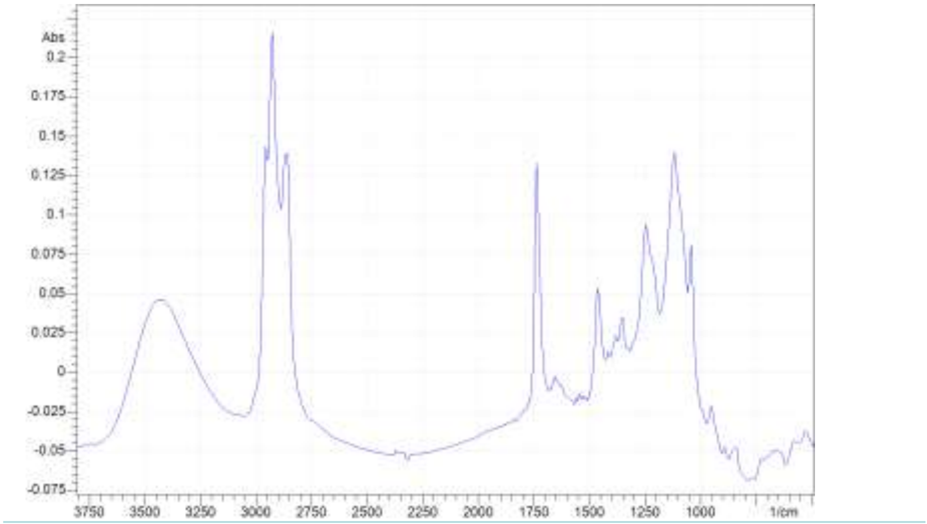
Real-Time, Portable, Field Analysis

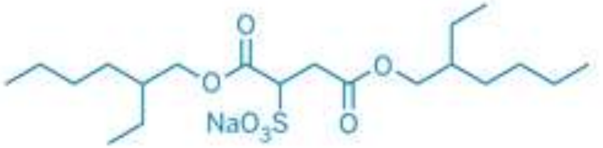
Analysis of Ocean Floor



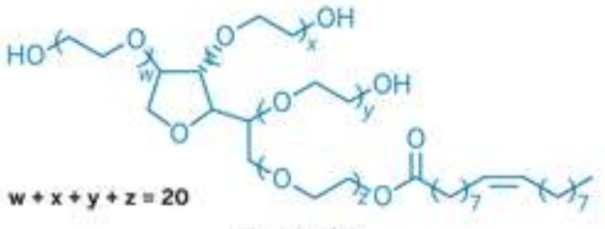
Identify oil/dispersants

DISPERSANT COREXIT 9527 on ZnSe



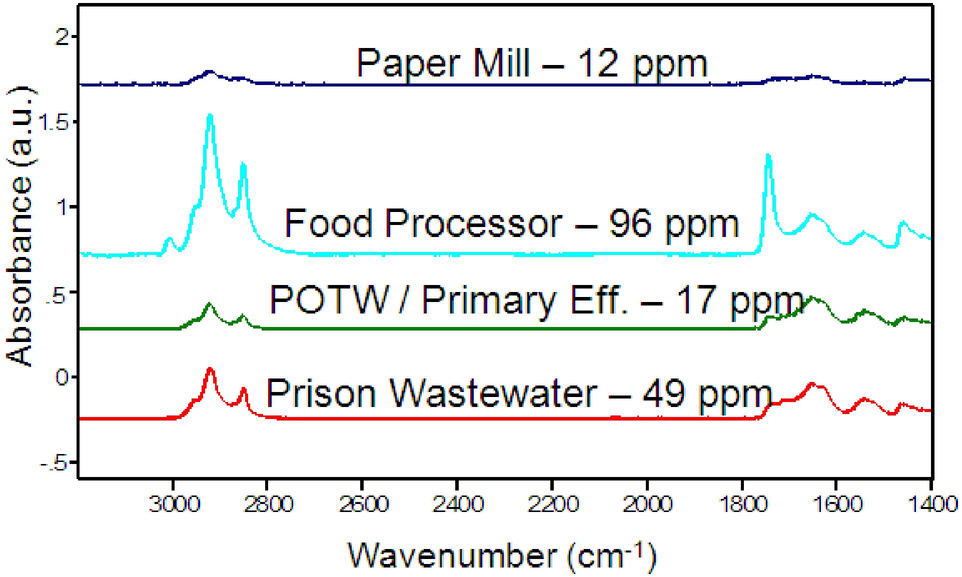


Sodium dioctyl sulfosuccinate



Tween 80

OTHER OIL AND GREASE MATRICES



- •STATISTICAL SIGNAL PROCESSING TO CHARACTERIZE / QUANTIFY DISPERSANTS IN OIL AND GREASE MATRICES??

- •CURRENTLY WORKING ON THIS.

AP Article 2/19/11

Scientist finds Gulf bottom still oily

'I've seen what it looks like with my own eyes. It's not going to be fine by 2012'

By SETH BORENSTEIN
Associated Press
updated 2/19/2011 11:41:15 PM ET

WASHINGTON — Oil from the BP spill remains stuck on the bottom of the Gulf of Mexico, according to a top scientist's

OSS ASTM D7575 Technology: USM Gulf Study

Objective: To study long-term effects of Deepwater Horizon disaster on marine mammals

Cruise #1: July 17-November 8

Cruise #2 (pending): Spring/summer 2011

OSS High Sensitivity Extractor

- Range: 0.2-40 ppm
- ML (As defined by EPA)= **200 ppb**

~75 Samples collected and processed through high-sensitivity OSS extractor

- Current results between non-detect and 0.6 ppm

OSS visit to NAVSEA Ship Systems Engineering Facility

- OSS sucessfully demonstrated the potential of ASTM Method D7575 for shipboard bilgewater applications at the NAVSEA facility in Philadelphia, PA.

NAVSEA
NAVAL SEA SYSTEMS COMMAND

On-going Field Testing / Tier I Study: Cleveland Ohio

Southerly Wastewater Treatment Plant

The Southerly Wastewater Treatment Plant (WWTP) is located in the village of Cuyahoga Heights, Ohio. It is situated on a 273-acre site and serves a population of more than 601,000 in the Greater Cleveland area. This facility is the largest of the District's three wastewater treatment plants and on

more...

MISCELLANEOUS

HOMOGENIZATION EXAMPLE
(motor oil in water - sonication)

T = 0 min

T = 10 min

T = 20 min

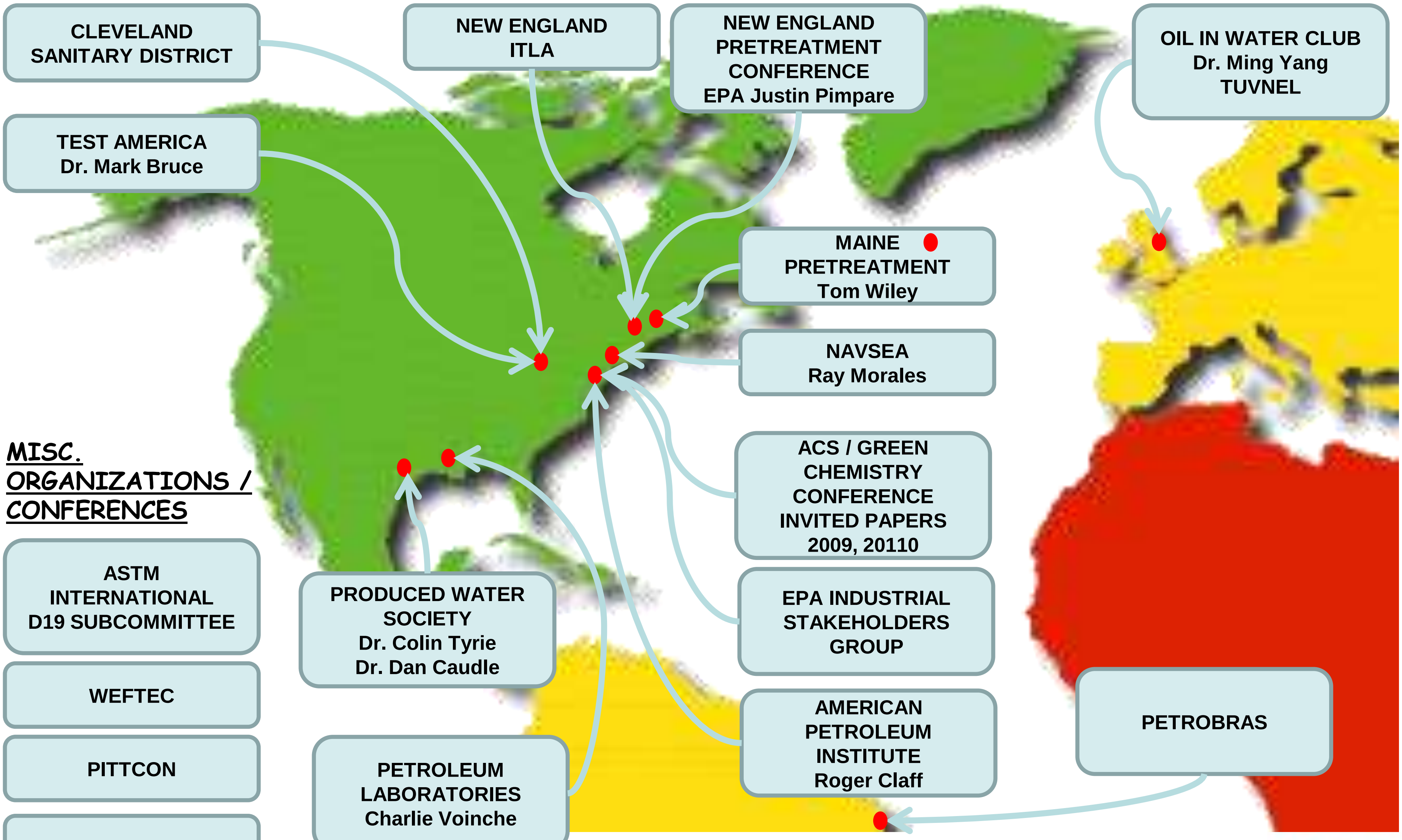
HOMOGENIZATION EXAMPLE (Bacon Grease in water - sonication)

T = 0 min

T = 10 min

T = 40 min

OSS OIL IN WATER INTERACTIONS



CLEVELAND
SANITARY DISTRICT

TEST AMERICA
Dr. Mark Bruce

NEW ENGLAND
ITLA

NEW ENGLAND
PRETREATMENT
CONFERENCE
EPA Justin Pimpare

OIL IN WATER CLUB
Dr. Ming Yang
TUVNEL

MAINE
PRETREATMENT
Tom Wiley

NAVSEA
Ray Morales

ACS / GREEN
CHEMISTRY
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2009, 20110

EPA INDUSTRIAL
STAKEHOLDERS
GROUP

AMERICAN
PETROLEUM
INSTITUTE
Roger Claff

PETROBRAS

PRODUCED WATER
SOCIETY
Dr. Colin Tyrie
Dr. Dan Caudle

PETROLEUM
LABORATORIES
Charlie Voinche

MISC. ORGANIZATIONS / CONFERENCES

ASTM
INTERNATIONAL
D19 SUBCOMMITTEE

WEFTEC

PITTCON

NELAC

Orono Spectral Solutions, Inc.
A Full Spectrum Innovator.

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- Incorporated 2004
- Spun out of University of Maine
- DoD Contractor (CBD-Army) for Chem/Bio Detection
- Actively Pursuing Commercial Opportunities