1

SCHEDULE

9:00 AM	INTRODUCTIONS
9:00 - 9:45 AM	SURVEY OF M313
9:45 AM- LUNCH	DEMONSTRATION [LAB]
1:00 - 2:30 PM	TELECONFERENCE [CC-2]
2:30 - 4:30 PM	PROCESSING/DISCUSSION [CC-2]
4:30 PM	WHOLE LAB TOUR [LAB]

2

INSTRUMENT CONFIGURATION

CALIBRATION

SAMPLE ANALYSIS

3

INSTRUMENT CONFIGURATION

4

CONSUMABLES

SEPTA, LINER, SYRINGE

SPLIT VS. SPLITLESS

CHROMATOGRAPHY VS EQUILIBRATION TIME

POST-COLUMN SPLIT

SPLITTER INSTALLATION

INSTRUMENT CONFIGURATION

5

IOM PREPARATION

PREP DEMONSTRATION

INTERPRETING RESULTS

INSTRUMENT RECONFIGURATION LOOP

INSTRUMENT OPTIMIZATION MIX

6

IOM Component	Evaluation Of
C6 - C15	Distribution of Molecular Weights to FID
BFB	Mass Spec Ionization [TO-15]
Triglyme	Instrument Sensitivity
EGDE (IS)	Injector Reliability
Ethylene Glycol	Separation from Internal Standard
Propylene Glycol	
Methyl Palmitate	Endpoint Retention Time

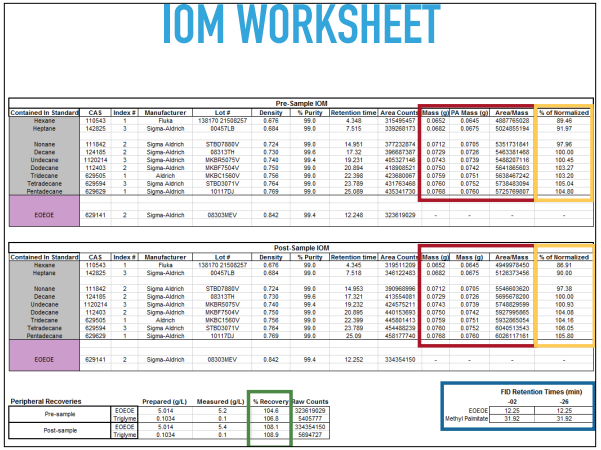
INSTRUMENT OPTIMIZATION MIX

7

IOM Component	QC Requirements
C6 - C15	Mass-adjusted area counts within 85 - 115% (normalized to C10)
BFB	Passes TO-15 Criteria
Triglyme	80 - 120% recovery at 0.1 g/L
EGDE (IS)	50 - 150% recovery
Ethylene Glycol	90% separation of each component (EG/EGDE/PG)
Propylene Glycol	
Methyl Palmitate	Within 0.1 min prior to and following sample analysis

IOM WORKSHEET

8



Pre-Sample IOM

Contained In Standard	CAS	Index #	Manufacturer	Lot #	Density	% Purity	Retention time	Area Counts	Mass (g)	PA Mass (g)	Area/Mass	% of Normalized
Hexane	110543	1	Fluka	1381702150825	0.676	99.0	4.348	315495457	0.0652	0.0645	4887765020	89.46
Heptane	142825	3	Sigma-Aldrich	00457LB	0.684	99.0	7.515	335268173	0.0682	0.0675	5024855194	91.97
Nonane	111842	2	Sigma Aldrich	STBD7880V	0.724	99.0	14.951	377232874	0.0712	0.0705	5351731841	97.96
Decane	124185	2	Sigma-Aldrich	68313TH	0.730	99.6	17.32	396687387	0.0729	0.0726	5463381466	100.09
Undecane	1120214	3	Sigma-Aldrich	MKBR5075V	0.740	99.4	19.231	405327146	0.0743	0.0739	5468207116	100.45
Dodecane	112403	2	Sigma-Aldrich	MKBF7504V	0.750	99.0	20.894	418905521	0.0750	0.0742	5641865693	103.27
Tridecane	629505	1	Aldrich	MKBC1560V	0.756	99.0	22.399	423680967	0.0759	0.0751	5638467242	103.29
Tetradecane	629594	3	Sigma Aldrich	STBD3071V	0.764	99.0	23.789	431763468	0.0760	0.0752	5738483094	105.04
Pentadecane	629629	1	Sigma-Aldrich	10117DJ	0.769	99.0	25.089	435341730	0.0768	0.0760	5725788987	104.80
EGEGE	629141	2	Sigma-Aldrich	08303MEV	0.842	99.4	12.248	323619629	-	-	-	-

Post-Sample IOM

Contained In Standard	CAS	Index #	Manufacturer	Lot #	Density	% Purity	Retention time	Area Counts	Mass (g)	Mass (g)	Area/Mass	% of Normalized
Hexane	110543	1	Fluka	1381702150825	0.676	99.0	4.345	319511269	0.0652	0.0645	4949978450	86.91
Heptane	142825	3	Sigma-Aldrich	00457LB	0.684	99.0	7.518	346122463	0.0682	0.0675	5126373456	90.00
Nonane	111842	2	Sigma Aldrich	STBD7880V	0.724	99.0	14.953	390968996	0.0712	0.0705	5546693620	97.38
Decane	124185	2	Sigma-Aldrich	68313TH	0.730	99.6	17.321	413554881	0.0729	0.0726	5695678200	100.00
Undecane	1120214	3	Sigma-Aldrich	MKBR5075V	0.740	99.4	19.232	424575213	0.0743	0.0739	5748829599	100.93
Dodecane	112403	2	Sigma-Aldrich	MKBF7504V	0.750	99.0	20.895	440153693	0.0750	0.0742	5927995065	104.08
Tridecane	629505	1	Aldrich	MKBC1560V	0.756	99.0	22.399	445801413	0.0759	0.0751	5932985054	104.16
Tetradecane	629594	3	Sigma Aldrich	STBD3071V	0.764	99.0	23.789	454488239	0.0760	0.0752	6040513543	106.05
Pentadecane	629629	1	Sigma-Aldrich	10117DJ	0.769	99.0	25.09	458177740	0.0768	0.0760	6026117161	105.80
EGEGE	629141	2	Sigma-Aldrich	08303MEV	0.842	99.4	12.252	334354150	-	-	-	-

Peripheral Recoveries

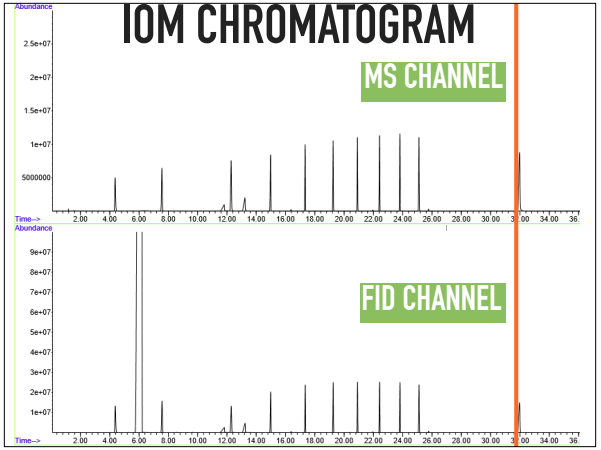
		Prepared (g/L)	Measured (g/L)	% Recovery	Raw Counts
Pre-sample	EGEGE	5.014	5.2	104.6	323619029
	Triglyme	0.1034	0.1	106.8	5405777
Post-sample	EGEGE	5.014	5.4	108.1	334354150
	Triglyme	0.1034	0.1	108.9	5894727

FID Retention Times (min)

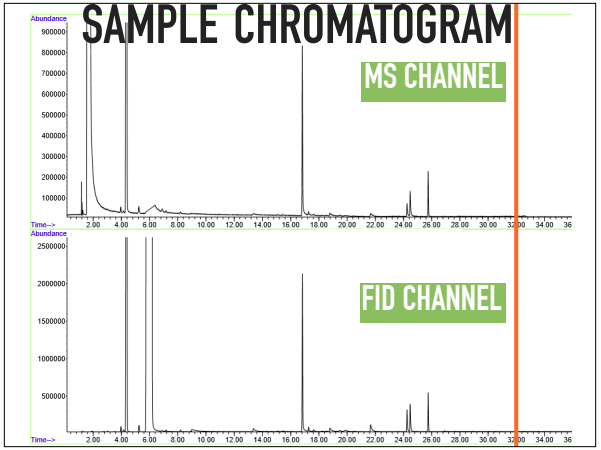
	-02	-26
EGEGE	12.25	12.25
Methyl Palmitate	31.92	31.92

IOM CHROMATOGRAM

9



SAMPLE CHROMATOGRAM



10

CONFIGURATION LOOP

1) Configure Instrument

2) IOM Preparation

3) IOM Analysis → Passing QC

11

CONFIGURATION LOOP

1) Tweak Configuration

2) IOM Preparation

3) IOM Analysis → Passing QC

x2

Failing QC

12

PRE-ANALYSIS CALIBRATION

13

REQUIRED BY EPA

THINGS TO CALIBRATE

A LIST, BY BRAD PARRACK

14

1) ???
2) ???
3) ???

DO YOU KNOW WHAT'S IN YOUR SAMPLE?

15

INDUSTRY LABS 

EVERYONE ELSE 

EVERYONE ELSE

SAMPLE PRE-SCREENING

1) IDENTIFY THE COMPOUNDS IN A SAMPLE
2) ESTIMATE THEIR CONCENTRATION TO DETERMINE IF A COMPOUND REQUIRES DIRECT CALIBRATION OR IF A SURROGATE CAN BE USED

ALERTS USER TO PRESENCE OF EXEMPT COMPOUNDS WHICH ARE QUANTIFIED ELSEWHERE AND REMOVED FROM THE VOC COATING CALCULATION

16

SAMPLE PRE-SCREENING

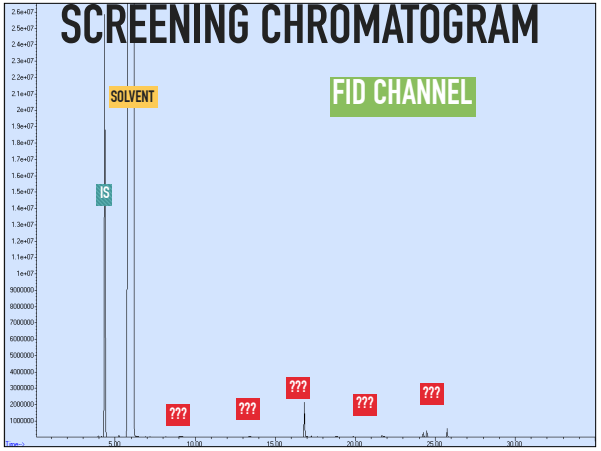
1)~ 3 G OF SAMPLE DILUTED INTO ~ 10 ML OF SOLVENT

2) 150 UL OF EGDE (IS) ADDED

3) MIXTURE BROUGHT TO 25 ML IN FLASK

17

SCREENING CHROMATOGRAM



18

DEFAULT COMPOUND

TRIGLYME

RESPONSE FACTOR USED TO QUANTIFY PEAKS SEEN:

1) DURING SCREENING

2) DURING QUANTITATIVE ANALYSIS FOR PEAKS <1 G/L

19

THINGS TO CALIBRATE

A LIST, BY BRAD PARRACK

1) TRIGLYME
2) HEXANE – PENTADECANE
3) ???

20

SAMPLE SPIKING QC

Extraction Markers
IPA
DUBA
Triglyme
Heptane

Calibrate

DETERMINE EXTRACTION EFFICIENCY IN SOLVENT

21

THINGS TO CALIBRATE
A LIST, BY BRAD PARRACK

1) TRIGLYME
2) HEXANE – PENTADECANE
3) IPA, HEPTANE, DIIBA

22

OVERHEARD IN THE LAB

CALIBRATION TIME!

An Excited Analyst

23

CALIBRATIONS CURVES

4 Point Calibration	2 Point Calibration
Surrogate Spikes	Most Compounds
Difficult Compounds	

24

CALIBRATIONS CURVES

25

4 Point Calibration	2 Point Calibration
0.1 g/L 1 g/L 5 g/L 15 g/L Forced Through Zero	0.1 g/L 15 g/L Forced Through Zero

26

4 Point Calibration	2 Point Calibration

Inj. #	Sample
1	Method Blank
2	IOM
3	Method Blank
4	15 g/L
5	Method Blank
6	15 g/L
7	Method Blank
8	5 g/L
9	Method Blank
10	5 g/L
11	Method Blank
12	1 g/L
13	Method Blank
14	1 g/L
15	Method Blank
16	0.1 g/L
17	Method Blank
18	0.1 g/L
19	Method Blank
20	IOM

Inj. #	Sample
1	Method Blank
2	IOM
3	Method Blank
4	15 g/L
5	Method Blank
6	15 g/L
7	Method Blank
8	0.1 g/L
9	Method Blank
10	0.1 g/L
11	Method Blank
12	IOM

27

CALIBRATION CURVES

2 POINT CURVE PREP – TRIGLYME & DIIBA

	15 g/L Standard		
	Addition	Mass (g)	g/L
25 ml Volumetric Flask (Class A)	-	46.8943	-
THF	~10 mL	61.6544	-
EOEOE	150 uL	61.7815	5.0535
From stock	-	-	-
DIIBA	397 uL	62.1671	15.2698
Triglyme	397 uL	62.5471	15.0024
QS with THF	To 25 mL	68.921	-

	0.1 g/L Standard		
	Diluted from 5 g/L standard		
	Addition	Mass (g)	g/L
25 ml Volumetric Flask (Class A)	-	46.9633	-
THF	~10 mL	59.7051	-
EOEOE	150 uL	59.8323	5.0575
From 5 g/L stock	500 uL	60.2856	-
DIIBA	-		0.1036
Triglyme	-		0.0977
QS with THF	To 25 mL	68.9922	-

FAMOUS MOVIE QUOTES

I FEEL THE NEED.
THE NEED TO SCREEN.

Tom Cruise

28

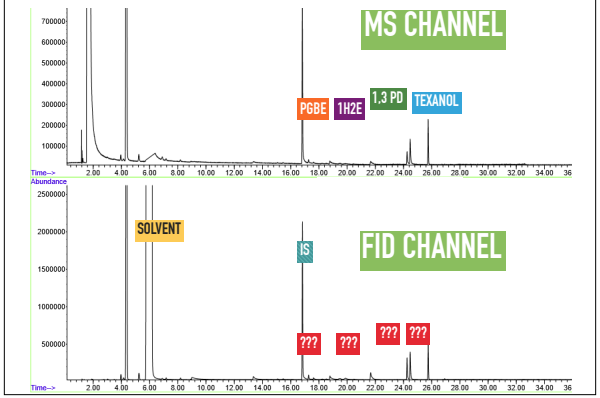
SAMPLE SCREENING SEQUENCE

Inj. #	Sample
1	Method Blank
2	IOM
3	Method Blank
4	0.1 g/L Exempts Standard
5	0.1 g/L Exempts Standard
6	Method Blank
7	Example Sample
8	Method Blank
9	Example Sample
10	Method Blank
11	0.1 g/L Exempts Standard
12	0.1 g/L Exempts Standard
13	Method Blank
14	IOM

6 SAMPLE MAX

29

SCREENING CHROMATOGRAM



30

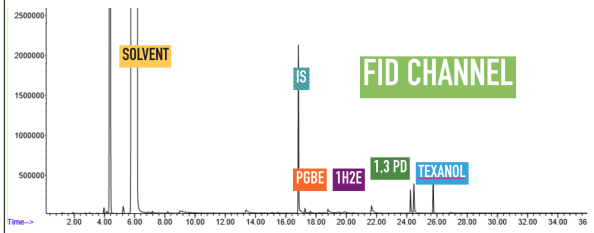
WHEN YOU NEED TO HAVE A RESPONSE FACTOR

Concentration (g/L)	Action
3+	Calibration Required
1 - 3	Calibration Required -or- Calibrate for Surrogate Required
0.1 - 1	Do Not Calibrate (quant as default compound)
< 0.1	Not counted in sample

Calibrate

31

SCREENING CHROMATOGRAM



32

SCREENING RESULTS AS TRIGLYME

-15.D	FID RT	CMPD Seen	~[g/L]
18	16.83	2-Propanol, 1-butoxy-	5.0
23	18.79	1-Hexanol, 2-ethyl-	0.4
26	21.67	1,3-Pentanediol, 2,2,4-trimet	0.7
27	24.25	Texanol	0.8
28	24.48	Texanol	1.2

REQUIRED CALIBRANT

REQUIRED CALIBRANT
-OR-
REQUIRED SURROGATE

QUANT AS DEFAULT

33

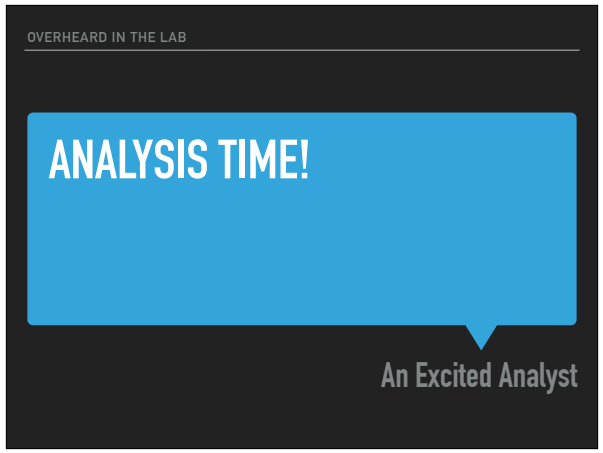
A NEW LIST OF THINGS TO CALIBRATE

A SECOND LIST, BY BRAD PARRACK

34

1) PGBE
2) TEXANOL

HAPPY CALIBRATING!



35

SAMPLE ANALYSIS

36

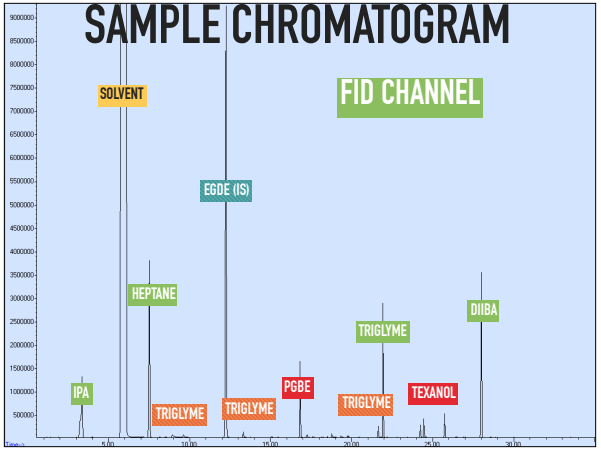
1) ~ 30 G OF SAMPLE SPIKED WITH 1 G/L OF EACH EXTRACTION MARKER [IPA/DIIBA/HEPANE/TRIGLYME]

2) ~ 3 G OF SAMPLE DILUTED INTO ~ 10 ML OF SOLVENT

3) 150 UL OF EGDE (IS) ADDED

4) MIXTURE BROUGHT TO 25 ML IN FLASK

SAMPLE CHROMATOGRAM



37

QUALITY CONTROLS STANDARDS

Continuing Spike Verification CSV	Continuing Cal Verification CCV
Monitors Spike Compounds for Cal Drift	Monitors Target Compounds for Cal Drift
EGDE (IS)	EGDE (IS)
Heptane	PGBE
Triglyme	Texanol
IPA	Target Compounds From Other Samples in Sequence
DIIBA	

38

SAMPLE ANALYSIS SEQUENCE

Inj. #	Sample
1	Method Blank
2	IOM
3	Method Blank
4	CSV
5	CSV
6	Method Blank
7	CCV
8	Method Blank
9	CCV
10	Method Blank
11	Example Sample #1
12	Method Blank
13	Example Sample #1
14	Method Blank
15	Example Sample #2
16	Method Blank
17	Example Sample #2
18	Method Blank
19	CCV
20	Method Blank
21	CCV
22	Method Blank
23	CSV
24	CSV
25	Method Blank
26	IOM

3 SAMPLE MAX

39

Required or Flexible?

40

Flexible	Required
Post Column Split, Two Column System, or Two Discrete Instruments	G43 Column Phase
	Column Temp Limit > 300 °C
	Compound Calibration Prior To Analysis
Solvent (MeOH vs THF)	
Consumables	
Split Vs. Splitless	
Column Dimensions	

EASY AS 1,2,3 . . . 4,5,6,7,8,9

41

1) CONFIGURE INSTRUMENT
2) PREP IOM; EVALUATE RESULTS
3) RECONFIGURE INSTRUMENT IF NECESSARY
4) CALIBRATE DEFAULT COMPOUNDS AND SPIKES
5) SCREEN SAMPLES IF UNKNOWN
6) CALIBRATE SCREENED/KNOWN COMPOUNDS AND SURROGATES FROM 1+ G/L (CALCULATED AS TRIGLYME)
7) PREPARE CCV, CSV; SPIKE/DILUTE SAMPLE
8) ANALYZE ON INSTRUMENT
9) PROCESS RESULTS

42

ME, RIGHT NOW

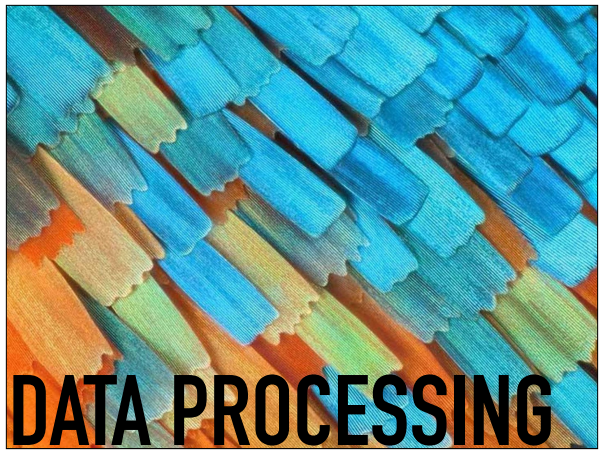
DEMONSTRATION TIME!

Brad Parrack

TOUR GROUPS

43

Brad	Tereso	Hanna
David Darling	Stephen Foster	Kristy Rodriguez
Dave Nevisson	Gina Johnson	Kimberly Gutierrez
Chrissy Ford	Brian Morehouse	Jerry Powers
Cathy Willis	Chris Pollack	Chris Nardi
Cidnie Hoang	Barry Cupp	Barry Marcks



44

DEVELOPING A CALIBRATION LIBRARY

45

CALCULATE RESPONSE FACTORS FOR:

- Internal Standard
- Target Compounds

EACH SOLVENT REQUIRES ITS OWN SET OF CALIBRANTS

46

4 Point Calibration

Inj. #	Sample
1	Method Blank
2	IOM
3	Method Blank
4	15 g/L
5	Method Blank
6	15 g/L
7	Method Blank
8	5 g/L
9	Method Blank
10	5 g/L
11	Method Blank
12	1 g/L
13	Method Blank
14	1 g/L
15	Method Blank
16	0.1 g/L
17	Method Blank
18	0.1 g/L
19	Method Blank
20	IOM

2 Point Calibration

Inj. #	Sample
1	Method Blank
2	IOM
3	Method Blank
4	15 g/L
5	Method Blank
6	15 g/L
7	Method Blank
8	0.1 g/L
9	Method Blank
10	0.1 g/L
11	Method Blank
12	IOM

47

INTERNAL STANDARD RESPONSE

Retention Time	Area Counts	IS Prepared (g/L)
12.272	366039509	5.053
12.271	367831644	5.053
12.27	368661304	5.057
12.272	370512860	5.057

Prepared concentrations and area counts compiled for internal standard

Response Factor for Internal Standard:

$$\frac{\sum \text{Prep'd Concentration} * \text{IS Area}}{\sum \text{Prep'd Concentration}^2}$$

Values are tabulated for Concentrations and Area Counts

	X*Y	X^2
	1849779195	25.53782
	1858835742	25.53782
	1864494222	25.57802
	1873858415	25.57802
Sum	**7446967574**	**102.2317**

48

INTERNAL STANDARD RESPONSE

Response Factor for Internal Standard:

$$\frac{7446967574}{102.2317} = 72843931$$

Response factor applied to area counts to determine percent recoveries of each injection.

Retention Time	Area Counts	Measured g/L	Prepared (g/L)	Percent Recovery
12.272	366039509	5.02	5.053	99.4
12.271	367831644	5.05	5.053	99.9
12.27	368661304	5.06	5.057	100.1
12.272	370512860	5.09	5.057	100.6

TARGET COMPOUND RELATIVE RESPONSE

49

Prepared concentrations and area counts compiled for internal standard and target compound

Prepared (g/L)	Retention Time	Area Counts	IS Prepared (g/L)	IS Retention Time (min)	IS Area Counts
15.270	8.872	1563769174	5.053	12.272	366039509
15.270	8.872	1572094144	5.053	12.271	367831644
0.104	8.755	10723977	5.057	12.27	368661304
0.104	8.757	10758046	5.057	12.272	370512860

Relative Response Factor (RRF) for Target Compound:

$$\frac{\sum([\text{Target}]/[\text{IS}]) \times (\text{Target AC}/\text{IS AC})}{\sum([\text{Target}]/[\text{IS}])}$$

TARGET COMPOUND RELATIVE RESPONSE

50

Relative Response Factor for Target Compound:

$$\frac{25.82423}{18.26125} = 1.4142$$

	X*Y	X^2
	12.90877	9.130206
	12.91427	9.130206
	0.000596	0.000419
	0.000595	0.000419
Sum	**25.82423**	**18.26125**

RRF applied to area counts to determine percent recoveries of each injection.

Retention Time	Area Counts	Measured g/L	Prepared (g/L)	Percent Recovery
8.872	1563769174	15.27	15.270	100.0
8.872	1572094144	15.27	15.270	100.0
8.755	10723977	0.10	0.104	100.4
8.757	10758046	0.10	0.104	100.3

RELATIVE RESPONSE FACTORS

51

COMPOUND	RRF	CAL DATE
1) Triglyme	0.7579	03/15/16
2) DIIBA	1.3850	03/15/16
3) Heptane	2.0510	03/15/16
4) IPA	1.0586	04/06/16
5) Nonane	2.0814	04/06/16
6) Octanol	1.7614	05/17/16
7) 4-Heptanone	1.5783	05/17/16
8) Ethylene Glycol	0.5928	05/17/16
9) Propylene Glycol	0.8091	05/17/16
10) Dipropylene Glycol	0.8981	05/17/16

DETECTION LIMITS

52

Required for all compounds with a RRF lower than Triglyme's RRF

RELATIVE RESPONSE FACTORS

53

COMPOUND	RRF	LOD	DATE
1) Triglyme	0.7579	-	03/15/16
2) DIIBA	1.3850	-	03/15/16
3) Heptane	2.0510	-	03/15/16
4) IPA	1.0586	-	04/06/16
5) Nonane	2.0814	-	04/06/16
6) Octanol	1.7614	-	05/17/16
7) 4-Heptanone	1.5783	-	05/17/16
8) Ethylene Glycol	0.5928	0.004 G/L	05/17/16
9) Propylene Glycol	0.8091	-	05/17/16
10) Dipropylene Glycol	0.8981	-	05/17/16

QUALITY CONTROLS

54

MB METHOD BLANK

IOM INSTRUMENT OPTIMIZATION MIX

CSV CONTINUING SPIKE VERIFICATION

CCV CONTINUING CALIBRATION VERIFICATION

CONTINUING SPIKE VERIFICATION

55

CSV
Monitors Spike Compounds for Cal Drift
EGDE (IS)
Heptane
Triglyme
IPA
DIIBA

SPIKES PREPARED AT 1 G/L

PREPARED GRAVIMETRICALLY

CONTINUING SPIKE VERIFICATION

56

Step	Name	grams	net (g)	%Purity	g/L
Tare	Flask	47.0628			
solvent	THF	59.5599	12.4971	99.0	494.8852
CMPD 1	EOEOE	59.6893	0.1294	99.4	5.1449
CMPD 2	Triglyme	59.7148	0.0255	98.7	1.0067
CMPD 3	IPA	59.7386	0.0238	99.99	0.9519
CMPD 4	DIIBA	59.7658	0.0272	99.2	1.0793
CMPD 5	Heptane	59.7870	0.0212	99.0	0.8395
CMPD 6					
CMPD 7					
solvent	THF	68.9512	9.1642	99.0	362.9023

Preparation

CONTINUING SPIKE VERIFICATION

57

peak #	R.T. min	Start min	End min	PK TY	peak height	corr. area	corr. % max.	% of total
1	1.205	1.156	1.330	BV	8743	179832	0.00%	0.000%
2	1.369	1.330	1.596	VB	27721	334469	0.00%	0.000%
3	3.972	3.856	4.150	BB	32991	1196474	0.00%	0.002%
4	4.361	4.203	4.716	BB	10543689	379250076	0.52%	0.478%
5	5.256	5.143	5.436	BV	34846	1786854	0.00%	0.002%
6	6.103	5.436	6.654	PV	622582593	73322864611	100.00%	92.492%
7	6.700	6.654	6.983	VB	6205	505744	0.00%	0.001%
8	7.161	6.996	7.390	BB	21757	1053989	0.00%	0.001%
9	7.539	7.403	7.996	BB	11244200	393560387	0.54%	0.496%
10	8.183	8.090	8.383	BB	15887	577742	0.00%	0.001%
11	8.530	8.410	8.743	BB	38743	2303056	0.00%	0.003%
12	9.628	9.536	9.916	BB	58490	2274521	0.00%	0.003%
13	10.420	10.303	10.650	BB	18425	871990	0.00%	0.001%
14	11.536	11.403	11.616	PV	10553367	429166155	0.59%	0.541%
15	11.751	11.616	12.136	VV	2109407	161329869	0.22%	0.204%
16	12.268	12.136	12.563	VV	9821362	363624514	0.50%	0.459%
17	12.635	12.563	12.870	VB	11579	713796	0.00%	0.001%
18	12.961	12.870	13.007	BV	9911	311885	0.00%	0.000%
19	13.190	13.007	13.596	VV	3826632	224410871	0.31%	0.283%
20	13.655	13.596	13.730	VV	16078	500239	0.00%	0.001%
21	14.963	14.829	15.144	PV	15462512	421896327	0.58%	0.532%
22	15.415	15.228	15.482	VV	9546	626386	0.00%	0.001%
23	15.538	15.482	15.670	VV	7208	476384	0.00%	0.001%
24	15.718	15.670	15.808	VV	7232	270856	0.00%	0.000%
25	15.949	15.808	16.110	VV	13328	520755	0.00%	0.001%

Results From Instrument

CONTINUING SPIKE VERIFICATION

58

RT	Area	Confirmation	Vlookup RF	final VOC g/L
1.19	221335	default	0.758	0.00
1.35	461406	default	0.758	0.00
1.77	151344	default	0.758	0.00
3.36	64809430	default	0.758	0.01
6.06	65579808737	solvent	0.000	0.00
6.86	742690	default	0.758	0.00
7.14	974715	default	0.758	0.00
7.51	121777562	default	0.758	0.01
8.16	538005	default	0.758	0.00
8.51	668143	default	0.758	0.00
9.61	2296732	default	0.758	0.00
12.23	343765372	default	0.758	0.04
17.22	487029	default	0.758	0.00
17.60	366312	default	0.758	0.00
19.78	169408	default	0.758	0.00
21.93	54408231	default	0.758	0.01
22.17	247001	default	0.758	0.00
24.60	467552	default	0.758	0.00
25.74	14096929	default	0.758	0.00
26.45	614906	default	0.758	0.00
28.00	103473809	default	0.758	0.01
28.57	335362	default	0.758	0.00

Compiled Peak Data

CONTINUING SPIKE VERIFICATION

59

RT	Area	Confirmation	Vlookup RF	final VOC g/L
1.19	221335	default	0.758	0.00
1.35	461406	default	0.758	0.01
1.77	151344	default	0.758	0.00
3.36	64809430	IPA	1.059	0.92
6.06	65579808737	solvent	0.000	0.00
6.86	742690	default	0.758	0.01
7.14	974715	default	0.758	0.02
7.51	121777562	Heptane	2.051	0.89
8.16	538005	default	0.758	0.01
8.51	668143	default	0.758	0.01
9.61	2296732	default	0.758	0.05
12.23	343765372	EGDE	6.17E+7	5.57
17.22	487029	default	0.758	0.01
17.60	366312	default	0.758	0.01
19.78	169408	default	0.758	0.00
21.93	54408231	Triglyme	0.758	1.07
22.17	247001	default	0.758	0.00
24.60	467552	default	0.758	0.01
25.74	14096929	default	0.758	0.28
26.45	614906	default	0.758	0.01
28.00	103473809	DIBA	1.400	1.11
28.57	335362	default	0.758	0.01

Apply RRFs to Appropriate Peaks

CONTINUING SPIKE VERIFICATION

60

CSV Results			
Compound	Prepared	Measured	% Recovery
EGDE (IS)	5.14	5.57	108
Heptane	0.84	0.89	106
Triglyme	1.00	1.07	107
IPA	0.95	0.92	97
DIIBA	1.08	1.11	103

QC Allowance: 90 - 110%

CONTINUING CALIBRATION VERIFICATION

61

CCV should possess target compounds from all samples in the sequence

Same data processing concept as CSV

QC Allowance: 85 - 115%

SAMPLE PROCESSING

62

Step	Name	CAS (required for CMPDs)	weight (g)	net (g)	% Purity	Final wt fx
tare	vial		28.9927			
sample (~35 g)	Sample		57.1552	28.1625	100	0.966
CMPD 1	TRIGLYME	112492	57.4048	0.2496	98.7	0.008
CMPD 2	IPA	67630	57.6621	0.2573	99.99	0.009
CMPD 3	DIIBA	141048	57.9040	0.2419	99.2	0.008
CMPD 4	HEPTANE	142825	58.1513	0.2473	99.0	0.008

Sample Spiking

SAMPLE PROCESSING

63

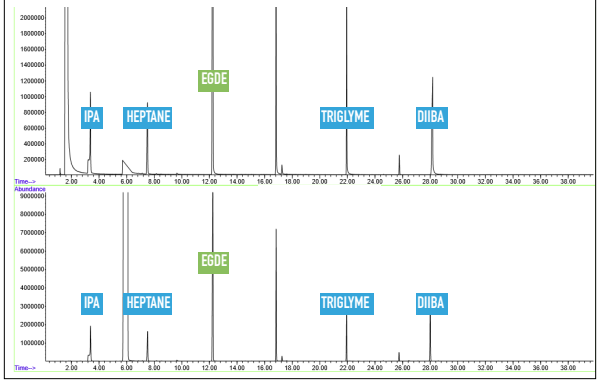
Step	Name	weight (g)	net (g)	% Purity	Final wt fx	g/L
tare	25 ml flask	51.0738				
solvent	THF	61.6716	10.5978	99.0		
I.S.	EGDE	61.7981	0.1265	99.4	0.006	5.030
spiked sample		65.4544	3.6563	100.0	0.156	141.256
solvent	THF	73.6818	8.2274	99.0		

	Final wt fx	g/L
TRIGLYME	0.0014	1.236
IPA	0.0014	1.290
DIIBA	0.0013	1.204
HEPTANE	0.0014	1.228

Sample Dilution

SAMPLE PROCESSING

64



SAMPLE PROCESSING

65

peak #	R.T. min	Start min	End min	PK TY	peak height	corr. area	corr. % max.	% of total
1	1.205	1.156	1.330	BV	8743	179832	0.00%	0.000%
2	1.369	1.330	1.596	VB	27721	334469	0.00%	0.000%
3	3.972	3.856	4.150	BB	32991	1196474	0.00%	0.002%
4	4.361	4.203	4.716	BB	10543689	379250876	0.52%	0.478%
5	5.256	5.143	5.436	BV	34846	1786854	0.00%	0.002%
6	6.103	5.436	6.654	PV	622582593	73322864611	100.00%	92.492%
7	6.700	6.654	6.983	VB	6205	585744	0.00%	0.001%
8	7.161	6.996	7.390	BB	21757	1053989	0.00%	0.001%
9	7.539	7.403	7.996	BB	11244200	393560387	0.54%	0.496%
10	8.183	8.090	8.383	BB	15887	577742	0.00%	0.001%
11	8.530	8.410	8.743	BB	38743	2303056	0.00%	0.003%
12	9.628	9.536	9.916	BB	58490	2274521	0.00%	0.003%
13	10.420	10.303	10.650	BB	18425	871990	0.00%	0.001%
14	11.536	11.403	11.616	PV	10553367	429166155	0.59%	0.541%
15	11.751	11.616	12.136	VV	2109407	161329869	0.22%	0.204%
16	12.268	12.136	12.563	VV	9821362	363624514	0.50%	0.459%
17	12.635	12.563	12.870	VB	11579	713796	0.00%	0.001%
18	12.961	12.870	13.007	BV	9911	311885	0.00%	0.000%
19	13.190	13.007	13.596	VV	3826632	224410871	0.31%	0.283%
20	13.655	13.596	13.730	VV	16078	500239	0.00%	0.001%
21	14.963	14.829	15.144	PV	15462512	421896327	0.58%	0.532%
22	15.415	15.228	15.482	VV	9546	626386	0.00%	0.001%
23	15.538	15.482	15.670	VV	7208	476384	0.00%	0.001%
24	15.718	15.670	15.808	VV	7232	270856	0.00%	0.000%
25	15.949	15.808	16.110	VV	13328	520755	0.00%	0.001%

Sample Data

SAMPLE PROCESSING

66

Peak No	Spl RT	Spl Area	Confirmation	Vlookup RF
1	1.77	201680	default	0.758
2	1.92	429948	default	0.758
3	3.24	17363809	default	0.758
4	3.38	69339639	default	0.758
5	6.05	57107853064	default	0.758
6	6.87	929388	default	0.758
7	7.15	635243	default	0.758
8	7.51	55939593	default	0.758
9	8.17	570912	default	0.758
10	9.64	2128814	default	0.758
11	12.24	329099881	default	0.758
12	14.59	247283	default	0.758
13	15.06	225204	default	0.758
14	16.83	172711923	default	0.758
15	17.26	7591679	default	0.758
16	17.61	716721	default	0.758
17	21.94	64183259	default	0.758
18	22.18	359277	default	0.758
19	24.60	356708	default	0.758
20	25.74	11612778	default	0.758
21	26.45	750768	default	0.758
22	27.11	227350	default	0.758
23	28.00	96717162	default	0.758
24	28.58	313033	default	0.758

PEAK ASSESSMENT

All peaks assigned
Triglyme RRF initially

SAMPLE PROCESSING

67

Peak No	Spl RT	Spl Area	Confirmation	Vlookup RF	Meas VOC g/L	Spl VOC g/L	
1	1.77	201680	default	0.758	0.004	0.03	
2	1.92	429948	default	0.758	0.009	0.06	
3	3.24	17363809	IPA	1.059	**0.251**	**1.77**	
4	3.38	69339639	IPA	1.059	**1.001**	**7.07**	
5	6.05	57107853064	default	0.758	0.000	0.00	
6	6.87	929388	default	0.758	0.019	0.13	
7	7.15	635243	default	0.758	0.013	0.09	
8	7.51	167818779	Heptane	2.051	**1.251**	**8.85**	SPIKE ASSIGNMENT
9	8.17	570912	default	0.758	0.012	0.08	
10	9.64	2128814	default	0.758	0.043	0.30	
11	12.24	329099881	EGDE	6.17E+7	5.334	**37.69**	
12	14.59	247283	default	0.758	0.005	0.04	IS ASSIGNMENT
13	15.06	225204	default	0.758	0.005	0.03	
14	16.83	172711923	default	0.758	3.483	24.61	
15	17.26	7591679	default	0.758	0.153	1.08	
16	17.61	716721	default	0.758	0.014	0.10	
17	21.94	64183259	Triglyme	0.758	**1.294**	**9.14**	
18	22.18	359277	default	0.758	0.007	0.05	
19	24.60	356708	default	0.758	0.007	0.05	
20	25.74	11812778	default	0.758	0.238	1.68	
21	26.45	750768	default	0.758	0.015	0.11	
22	27.11	227350	default	0.758	0.005	0.03	
23	28.00	96717162	DIIBA	1.400	**1.056**	**7.46**	
24	28.58	313033	default	0.758	0.006	0.04	

SAMPLE PROCESSING

68

Sample Spike Results

Compound	Prepared	Measured	% Recovery
EGDE (IS)	5.03	5.33	106
Heptane	1.23	1.25	102
Triglyme	1.24	1.29	104
IPA	1.29	1.25	97
DIIBA	1.20	1.06	88

QC Allowance: 85 - 115%

SAMPLE PROCESSING

69

Pre Blank RT	Pre Blank AC	Pre CSV RT	Pre CSV AC	Peak No	Spl RT	Spl Area	Confirmation	Vlookup RF	Meas VOC g/L	Spl VOC g/L
1.77	176247	1.77	184072	1	1.77	201680	default	0.758	0.004	0.03
				2	1.92	429948	default	0.758	0.009	0.06
		3.22	17686801	3	3.24	17363809	IPA	1.059	**0.251**	**1.77**
		3.37	48640581	4	3.38	69339639	IPA	1.059	**1.001**	**7.07**
6.08	67951221220	6.07	67977122016	5	6.05	57107853064	default	0.758	0.000	0.00
		6.86	764954	6	6.87	929388	default	0.758	0.019	0.13
7.15	712058	7.14	1004002	7	7.15	635243	default	0.758	0.013	0.09
		7.51	129162718	8	7.51	55939593	Heptane	2.051	**0.417**	**2.95**
8.17	552248	8.16	560369	9	8.17	570912	default	0.758	0.012	0.08
9.61	2331069	9.61	2327567	10	9.64	2128814	default	0.758	0.043	0.30
12.24	353533505	12.23	353199810	11	12.24	329099881	EGDE	6.17E+7	5.334	37.69
				12	14.59	247283	default	0.758	0.005	0.04
				13	15.06	225204	default	0.758	0.005	0.03
				14	16.83	172711923	default	0.758	3.483	24.61
				15	17.26	7591679	default	0.758	0.153	1.08
17.60	1145014	17.60	1170358	16	17.61	716721	default	0.758	0.014	0.10
		21.94	56086829	17	21.94	64183259	Triglyme	0.758	**1.294**	**9.14**
		22.17	245218	18	22.18	359277	default	0.758	0.007	0.05
		24.60	478330	19	24.60	356708	default	0.758	0.007	0.05
25.74	14677563	25.74	14755520	20	25.74	11812778	default	0.758	0.238	1.68
		26.45	721685	21	26.45	750768	default	0.758	0.015	0.11
				22	27.11	227350	default	0.758	0.005	0.03
		28.00	105955275	23	28.00	96717162	DIIBA	1.400	**1.056**	**7.46**
		28.58	338987	24	28.58	313033	default	0.758	0.006	0.04

SAMPLE PROCESSING

70

Peak No	Spl RT	Spl Area	Confirmation	Vlookup RF	Spl VOC g/L
1	1.77	201680	solvent	0.000	0.00
2	1.92	429948	default	0.758	0.06
3	3.24	17363809	IPA	1.059	**1.77**
4	3.38	69339639	IPA	1.059	**7.07**
5	6.05	57107853064	solvent	0.000	0.00
6	6.87	929388	default	0.758	0.13
7	7.15	635243	solvent	0.000	0.00
8	7.51	55939593	Heptane	2.051	**2.95**
9	8.17	570912	solvent	0.000	0.00
10	9.64	2128814	solvent	0.000	0.00
11	12.24	329099881	EGDE	6.17E+7	**37.69**
12	14.59	247283	default	0.758	0.04
13	15.06	225204	default	0.758	0.03
14	16.83	172711923	default	0.758	24.61
15	17.26	7591679	default	0.758	1.08
16	17.61	716721	solvent	0.000	0.00
17	21.94	64183259	Triglyme	0.758	**9.14**
18	22.18	359277	default	0.758	0.05
19	24.60	356708	default	0.758	0.05
20	25.74	11812778	solvent	0.000	0.00
21	26.45	750768	default	0.758	0.11
22	27.11	227350	default	0.758	0.03
23	28.00	96717162	DIBA	1.400	**7.46**
24	28.58	313033	default	0.758	0.04

SAMPLE PROCESSING

71

Results

Compound Seen	Concentration as Triglyme	RRF Used	RRF	Final Concentration
PGBE	24.61	PGBE	1.179	15.82
2-ethyl hexanol	1.08	Octanol	0.84	0.48

SAMPLE PROCESSING

72

Peak No	Spl RT	Spl Area	Confirmation	Vlookup RF	Spl VOC g/L
1	1.77	201680	solvent	0.000	0.00
2	1.92	429948	default	0.758	0.06
3	3.24	17363809	IPA	1.059	**1.77**
4	3.38	69339639	IPA	1.059	**7.07**
5	6.05	57107853064	solvent	0.000	0.00
6	6.87	929388	default	0.758	0.13
7	7.15	635243	solvent	0.000	0.00
8	7.51	55939593	Heptane	2.051	**2.95**
9	8.17	570912	solvent	0.000	0.00
10	9.64	2128814	solvent	0.000	0.00
11	12.24	329099881	EGDE	6.17E+7	**37.69**
12	14.59	247283	default	0.758	0.04
13	15.06	225204	default	0.758	0.03
14	16.83	172711923	PGBE	1.179	15.82
15	17.26	7591679	Octanol	1.716	0.48
16	17.61	716721	solvent	0.000	0.00
17	21.94	64183259	Triglyme	0.758	**9.14**
18	22.18	359277	default	0.758	0.05
19	24.60	356708	default	0.758	0.05
20	25.74	11812778	solvent	0.000	0.00
21	26.45	750768	default	0.758	0.11
22	27.11	227350	default	0.758	0.03
23	28.00	96717162	DIBA	1.400	**7.46**
24	28.58	313033	default	0.758	0.04

SAMPLE PROCESSING

73

Final Sample Results (g/L)	
Toal VOC	82.62
Surrogate Spikes	66.08
Solvent (not counted)	0.32
Total Left As Default	0.24
Net VOC Material	16.54

SAMPLE PROCESSING

74

Final Sample Results	
Total Left As Default	0.24

Limit: 5 g/L or 10%; whichever is larger

SAMPLE PROCESSING SUMMARY

75

Spike Samples with Extraction Markers

Dilute Sample

Inject Sample

Import Data, Check Integrations, Assign Every Peak the Triglyme RRF

Assign RF to EOEOE peak; RRFs to Extraction Marker peaks

Remove Solvent Peaks from Summation

Identify Peaks > 1 g/L; Assign Direct RRF for Peaks > 3 g/L

Assign Direct RRF for Peaks > 1 and Less than 3 g/L -or- Assign Acceptable Surrogate RRF

Report Total VOC Material and QC for IOMs, CCVs, and CSVs

SURROGATE RULES

76

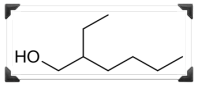
Between 1 - 3 g/L surrogate compounds may be utilized to quantify so long as they are within the following rules:

1) Surrogate compounds possess the same functional groups
2) Surrogate compounds are within 1 Carbon

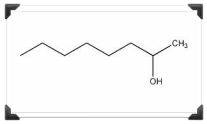
SURROGATE RULES

77

Example:
2-ethyl hexanol [2 g/L]



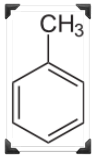
Acceptable Surrogate
Octanol



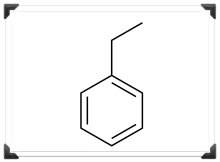
SURROGATE RULES

78

Example:
Toluene [1.5 g/L]



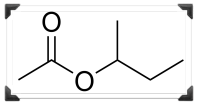
Acceptable Surrogate:
Ethyl Benzene



SURROGATE RULES

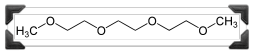
79

Example:
Butyl Acetate [0.75 g/L]



Acceptable Surrogate:

Triglyme
(< 1 g/L!)



ERROR BAND TREATMENT

80

Designed to limit error to
5 g/L VOC Material

ERROR BAND

81

EXAMPLE COATING

VOC Material:	20.8 g/L
Wt % Water:	45.6
NV %:	52.7
Density:	1.2 g/mL
VOC Coating:	**48.5 g/L**

ERROR BAND

VOC Material:	20.8 g/L
VOC Coating:	**48.5 g/L**
VOC Material (± 5 g/L):	15.8 g/L
	25.8 g/L

VOC Coating Range
37 - 60 g/L

82

83

84