Automation of Liquid-Liquid Extraction methodologies using an on-line Gerstel MPS with Agilent 5977B High Efficiency Source

Richard Davis
Applications Chemist - Anatune

Introduction

- Who are we?
- What we do?
- Automation of methods
- Summary

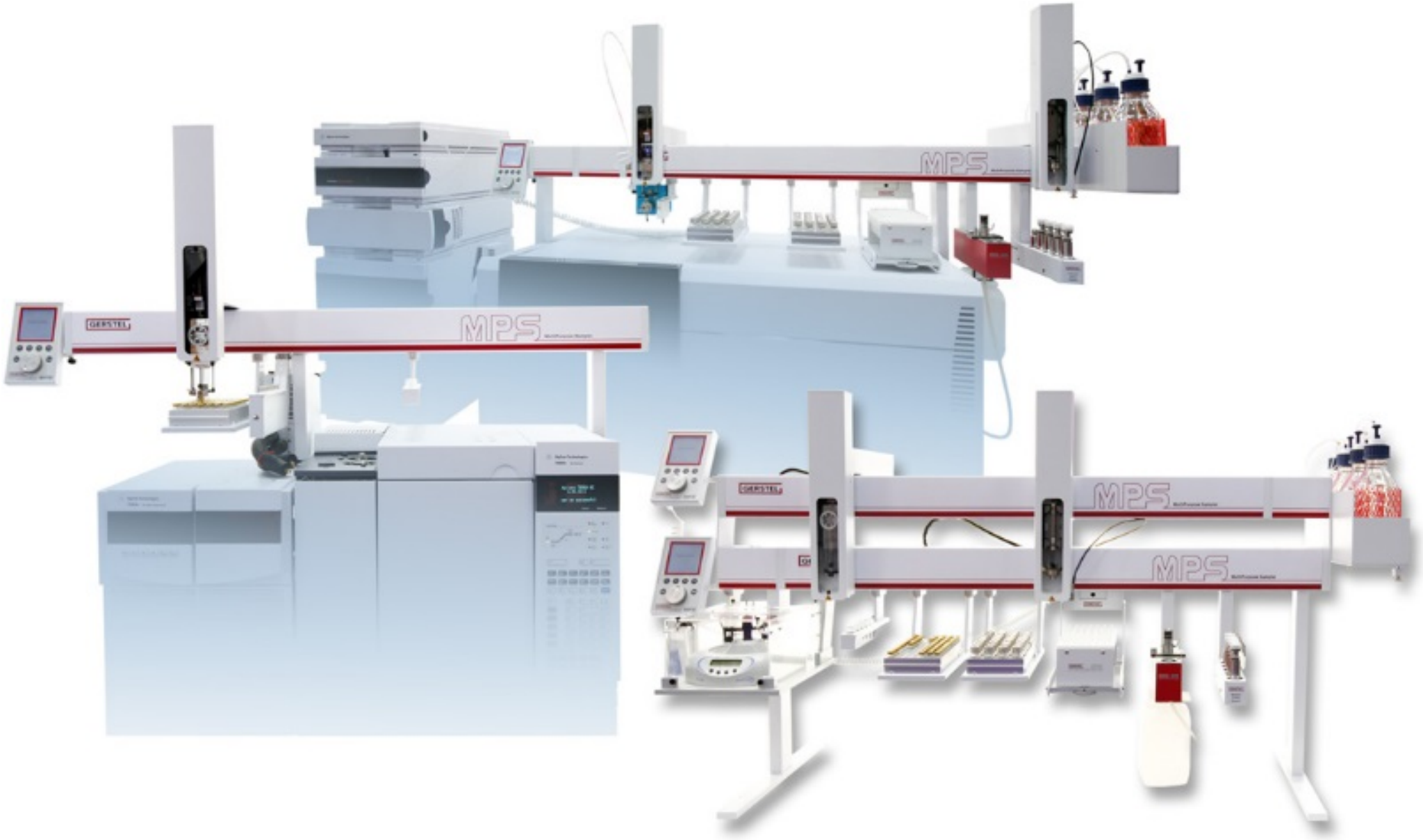
Who am I?

Who are Anatune?

Agilent Technologies

GERSTEL

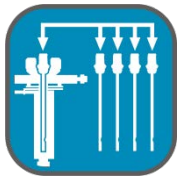
What we do - Solutions for Automation



Solutions for Automation


MultiPurpose Sampler MPS for GC/MS


Cooled Injection System CIS


Automated Liner EXchange ALEX


easy Liner Exchange eLEX


Opiate Solution


MultiFiber EXchange MFX


Thermal Desorption System TDS


Thermal Desorption Unit TDU


Automated TDU Liner Exchange ATEX


Solid Phase Microextraction SPME


Twister


Dynamic Headspace DHS

TDU PYRO


µFlowManager


Barcode Reader


Selectable 1D/2D GC/MS


Olfactory Detection Port OPD


Preparative Fraction Collector PFC


MultiPurpose Sampler MPS for LC/MS


Filtration


LC/MS Effluent Optimizer LEO


MPS Workstation


Solid Phase Extraction SPE


Disposable Pipette Extraction DPX


MultiPurpose Sampler MPS for LC/MS


MultiPosition Evaporation Station mVAP


MAESTRO PrepAhead


MAESTRO Software


THC Solution

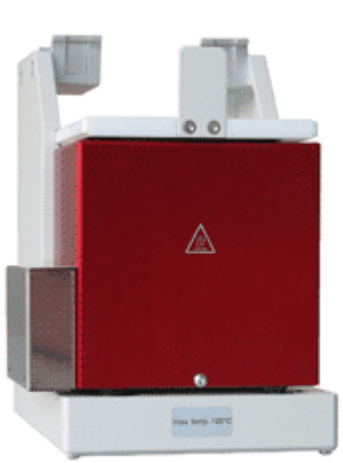

Agitation

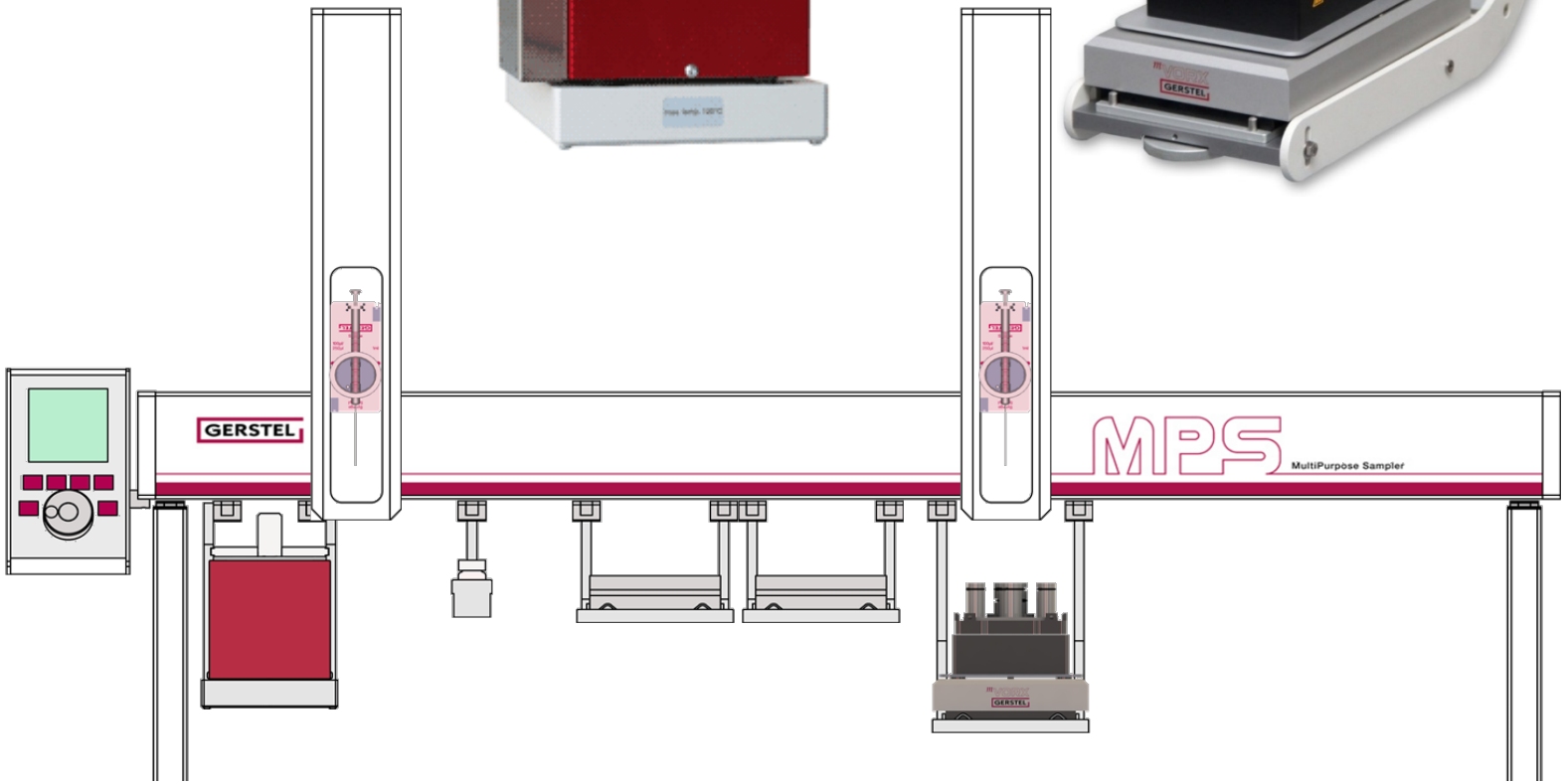
And more ...

Agitator and mVorx (mixing)

TASKS

- Homogenise/Mix
- 8 vials simultaneously
- 3000rpm





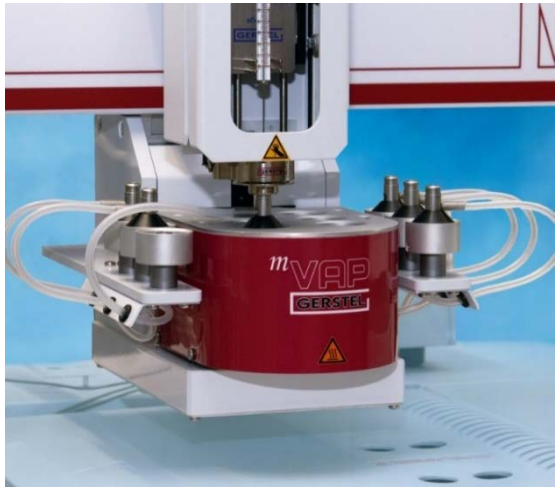
BENEFITS

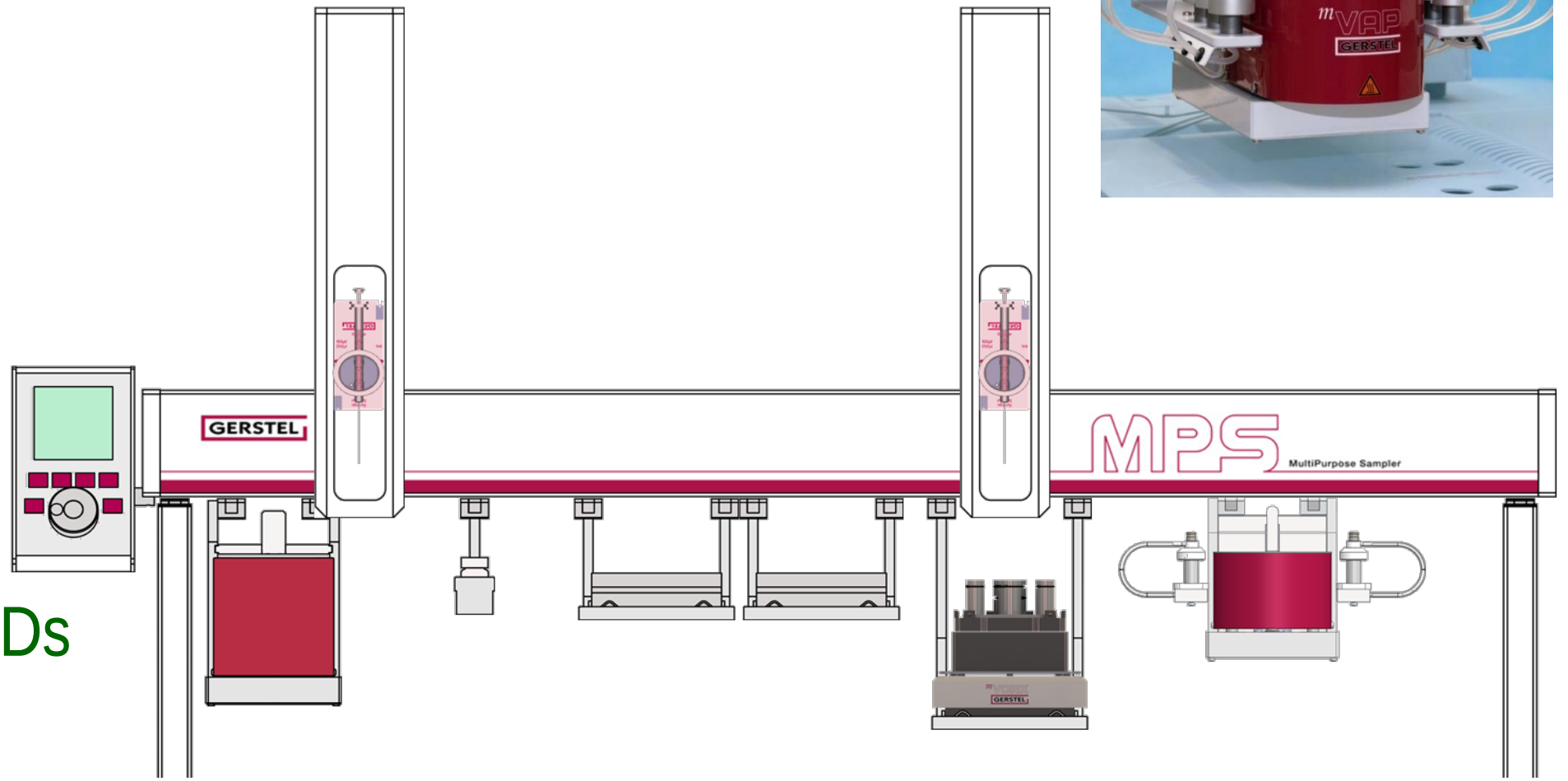
- Improved Extraction
- Precision
- Reproducibility
- Safety

mVap (Evaporate)

- Evaporate/Concentrate
- Derivatise
- Hydrolyse

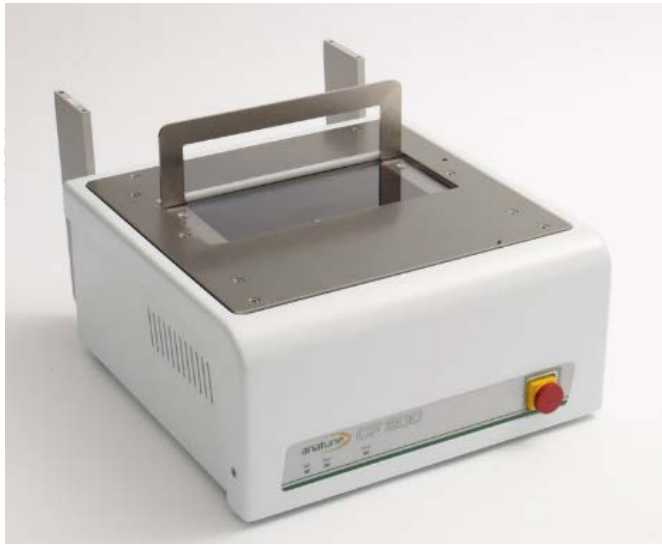
- Integrated
- Fast
- Safe
- Improved LODs





Centrifuge

- Separation
- Integrated
- Versitile

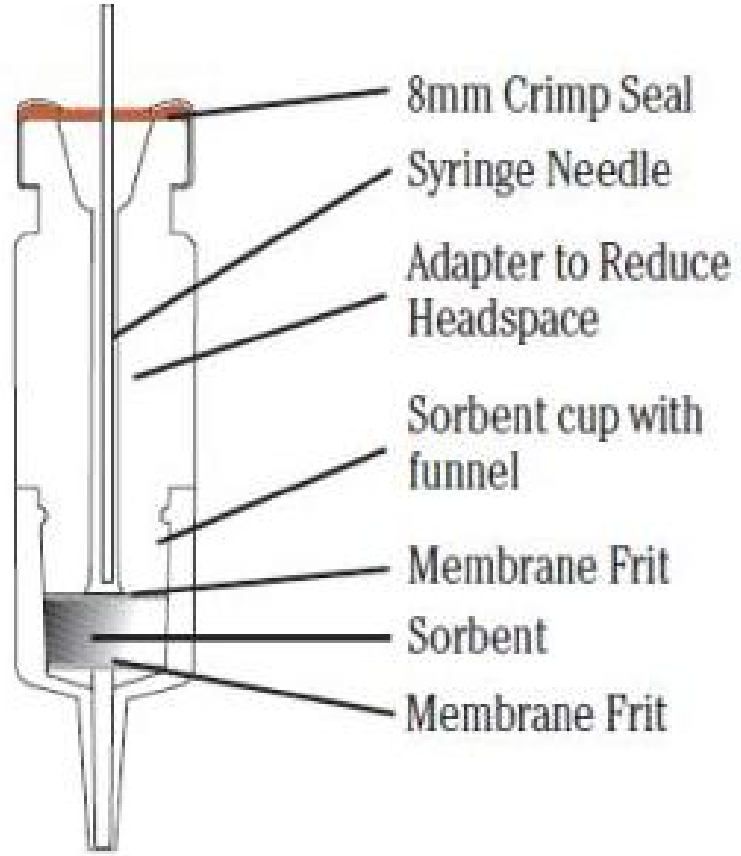


Solid Phase Extraction

- Concentration
- Selectivity
- Clean-up/Filtration
- Lower solvent cost
- Speed
- Safety

Instrument Top Sample Preparation

ITSP™ SOLUTIONS, INC.



Customers Existing Phenols Method

- 100mL Water
- Spike Cals and QCs
- Add 400µL Acid
- Shake
- Spike Surrogate
- Shake
- Add Salt
- Add Solvent/Derivatising agent
- Shake
- Add Base
- Wait 45 mins
- Adjust water level
- Add Salt
- Transfer solvent to GC vial
- Inject

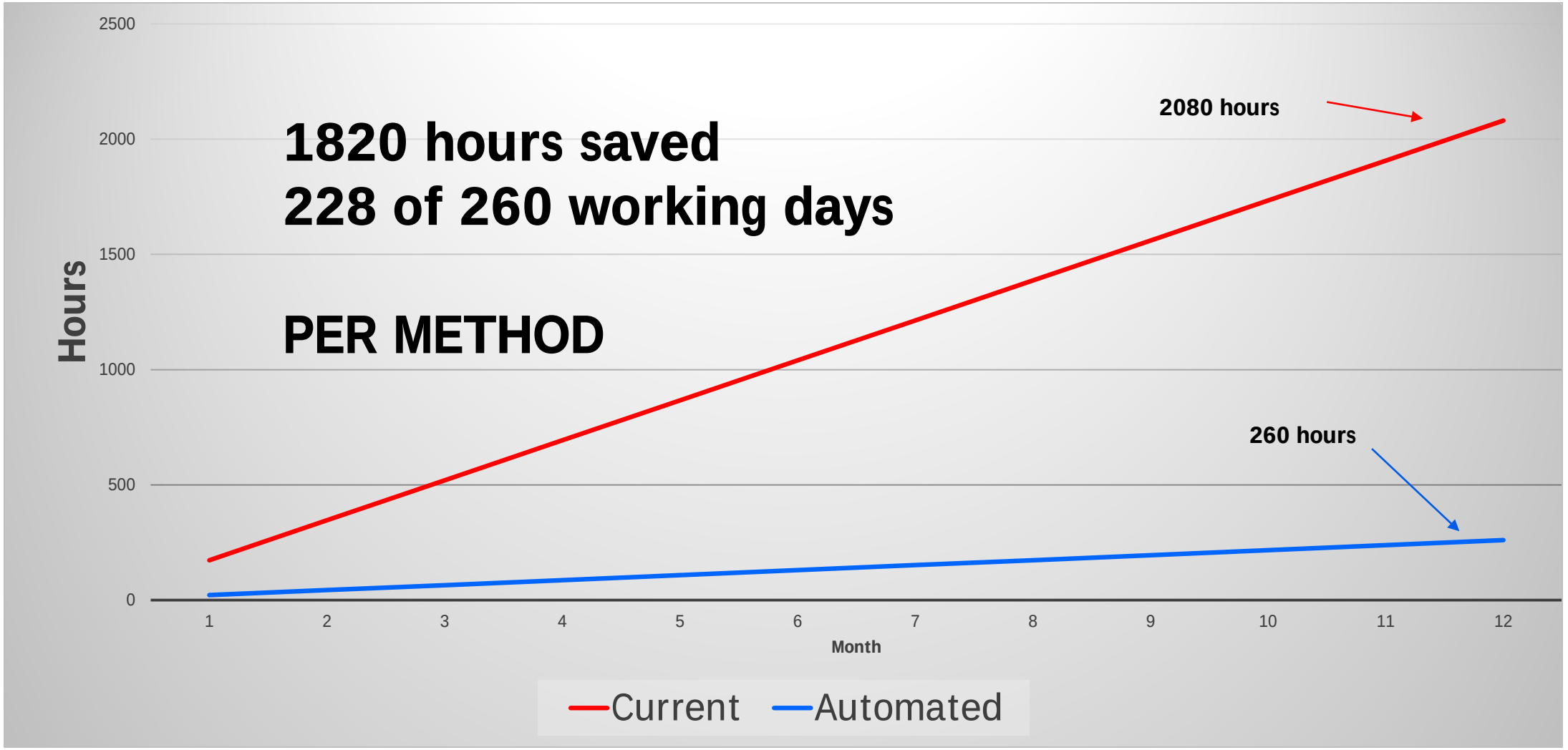
Phenols Automation

- 8mL Water (Manual)

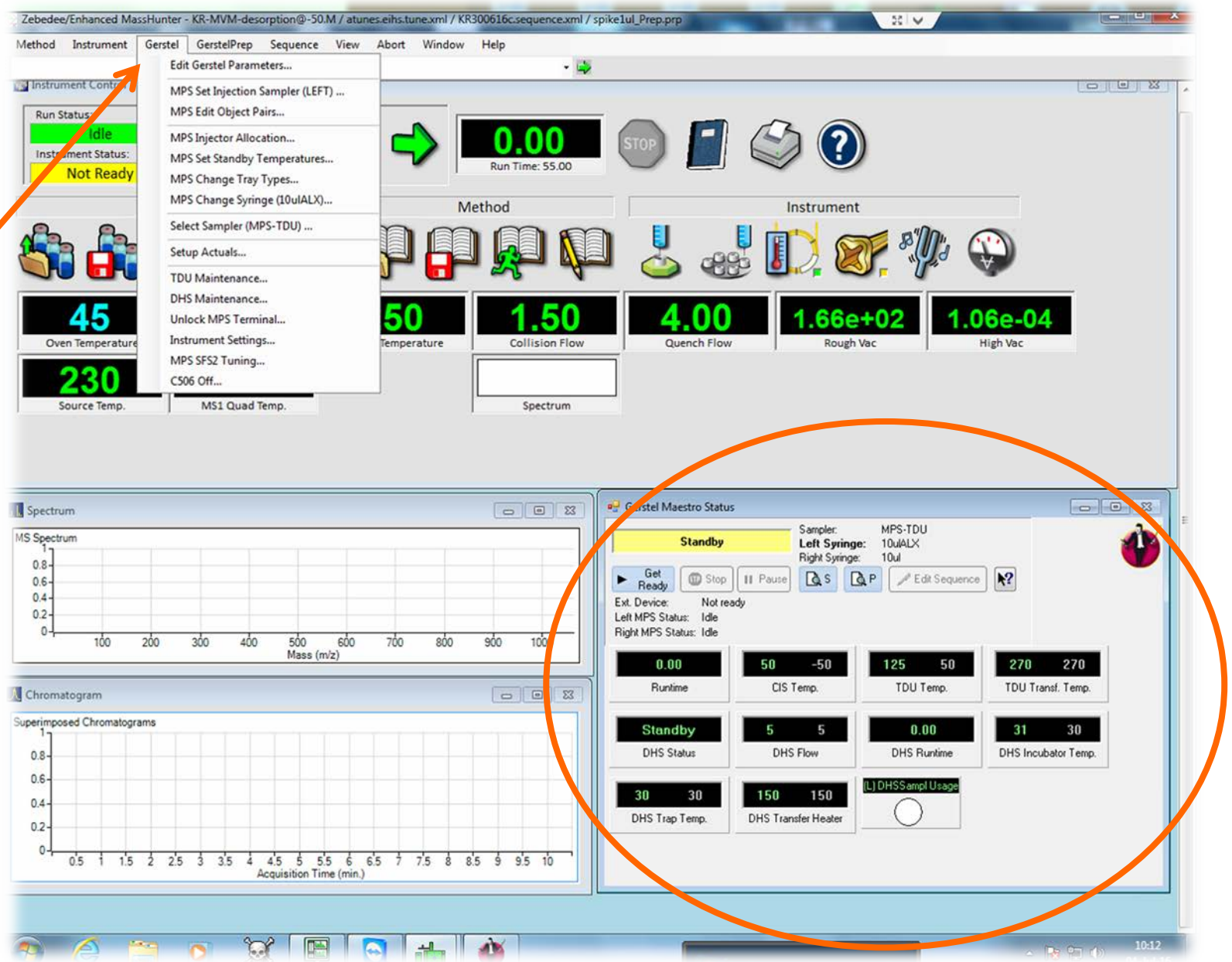
Automated;

- Add Acid
- Spike Cals and QCs & Surrogate
- Vortex
- Add Salt (aq)
- Vortex
- Add Solvent/Derivatising Agent
- Vortex
- Add Base
- Vortex
- Add 10µL Organic Solvent
- Inject from organic layer

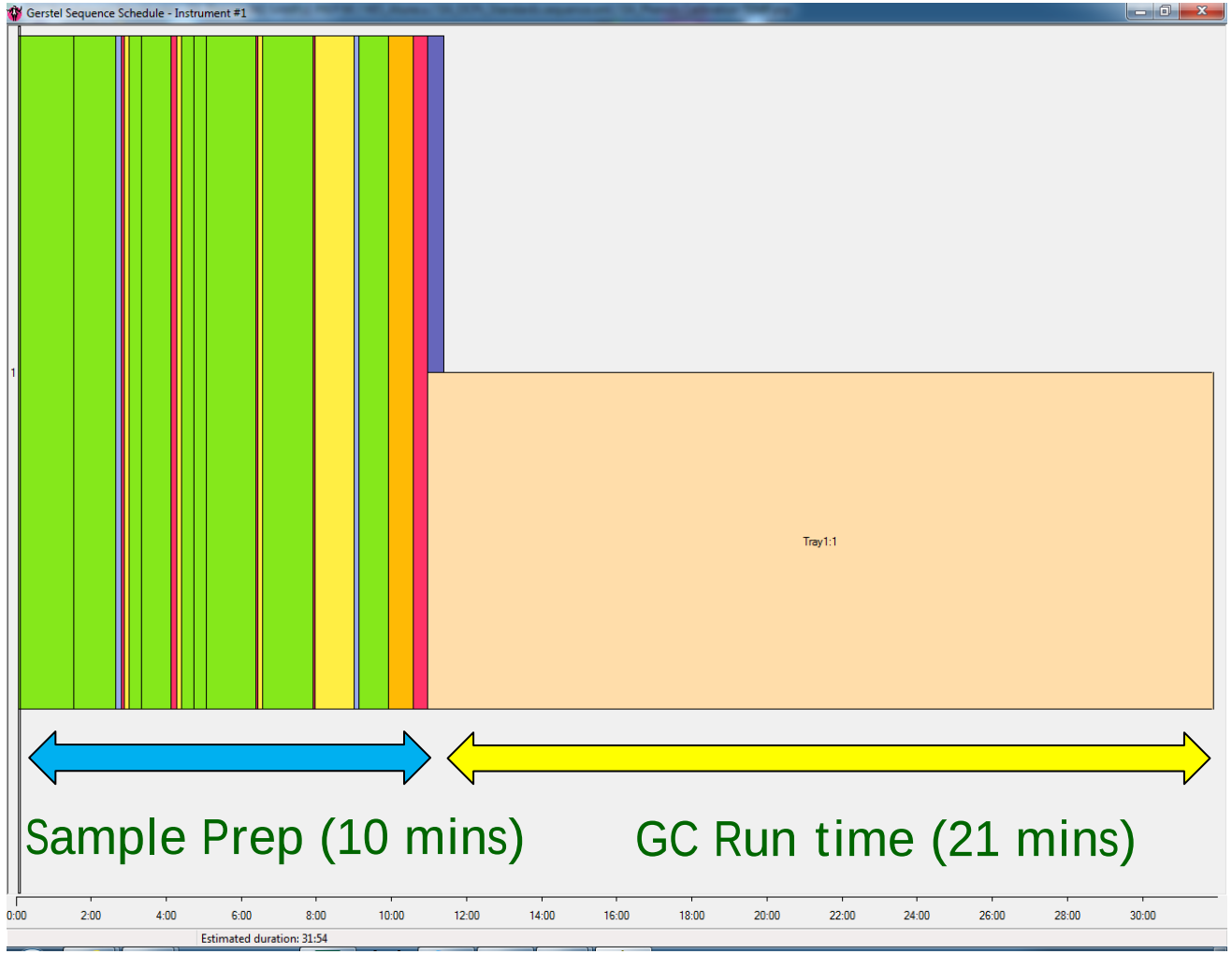
Sample Preparation Time – 1 year



Maestro and Mass Hunter



Automation of Phenols Sample Preparation

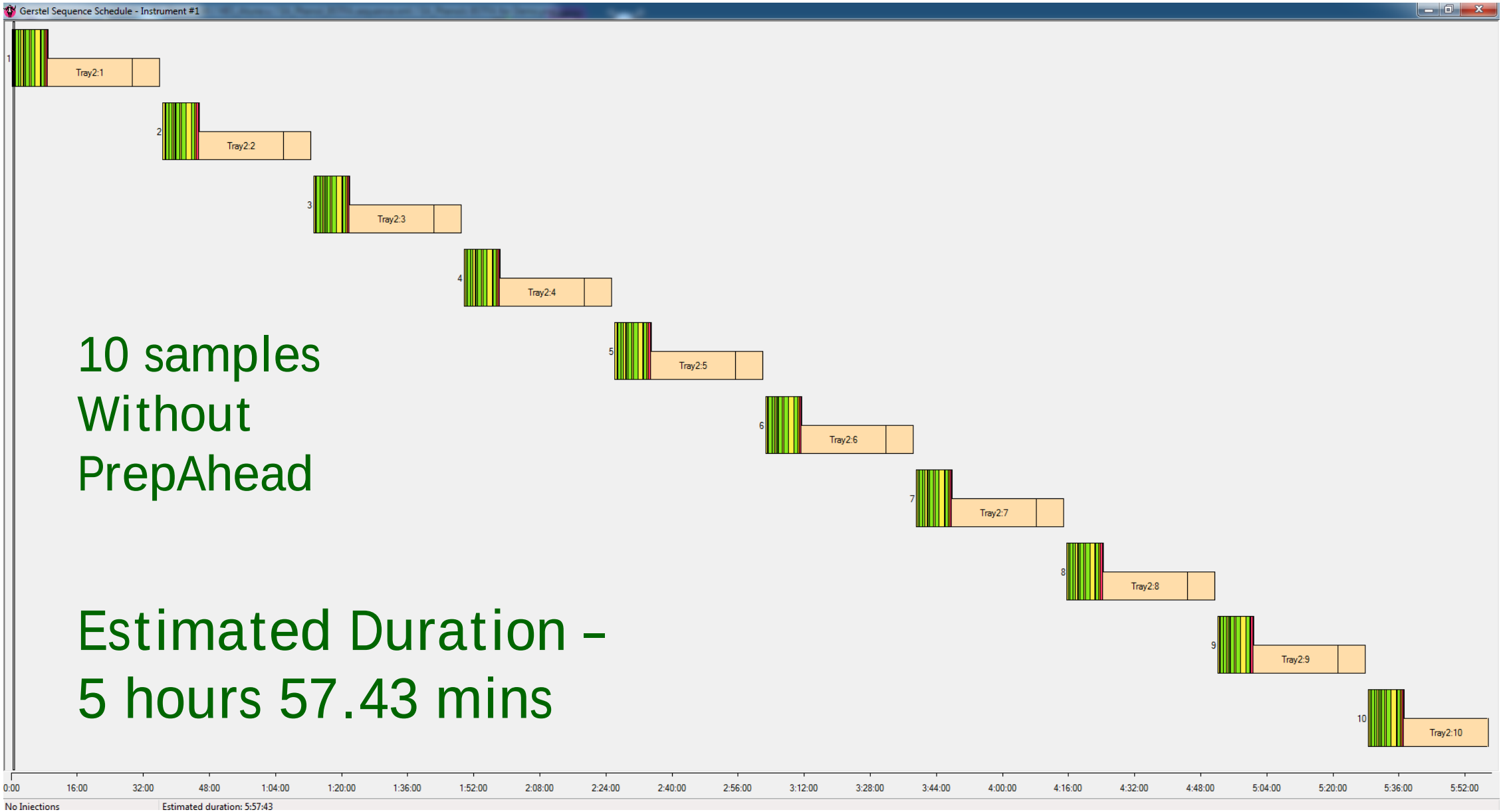


Sample Preparation

- Solvent Addition
- Syringe Washes
- Movement of vials
- Vortex mixing
- Injection to GC inlet

Estimated Duration – 31.54 mins

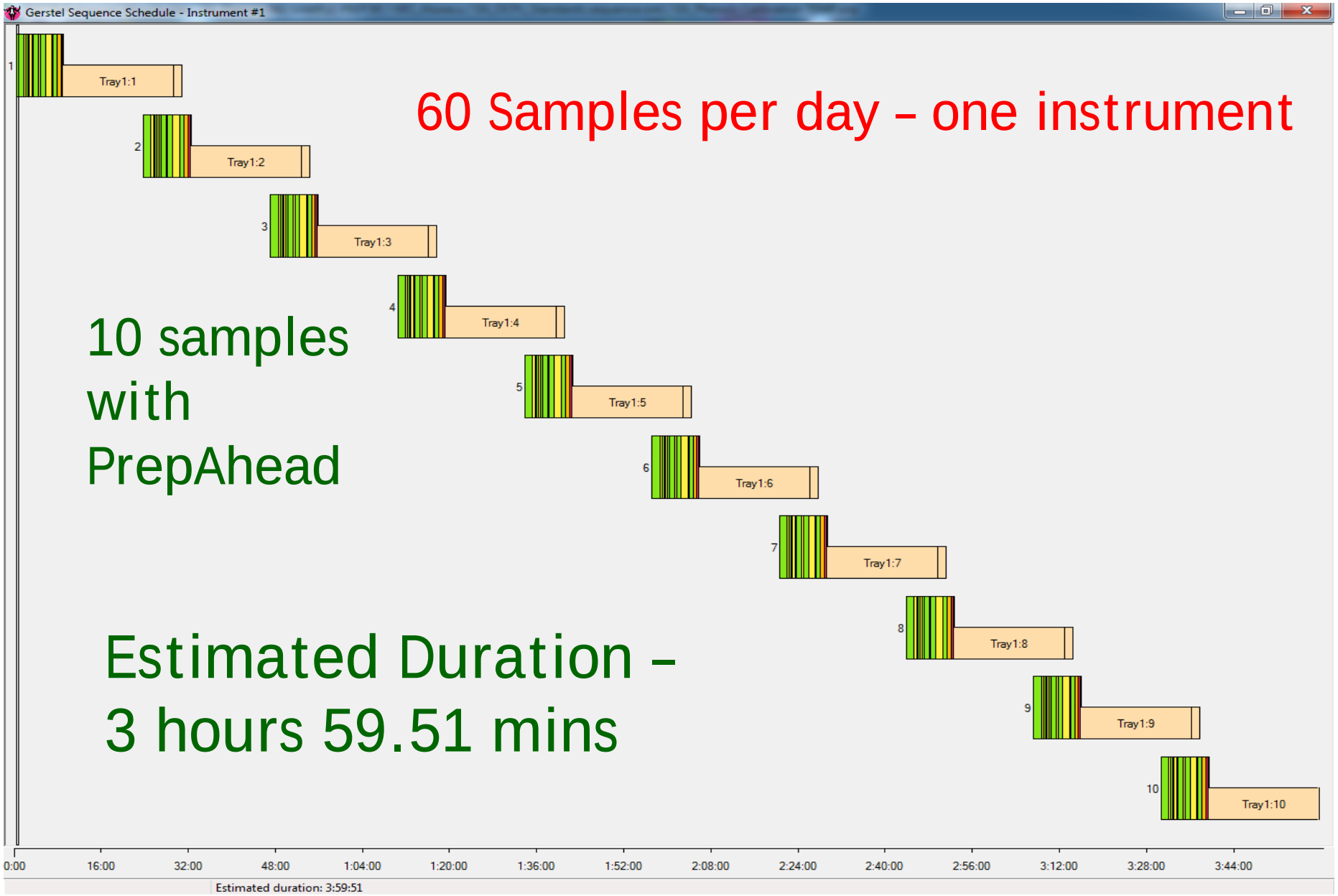
Phenols - No Prep Ahead



10 samples
Without
PrepAhead

Estimated Duration –
5 hours 57.43 mins

Phenols - PrepAhead

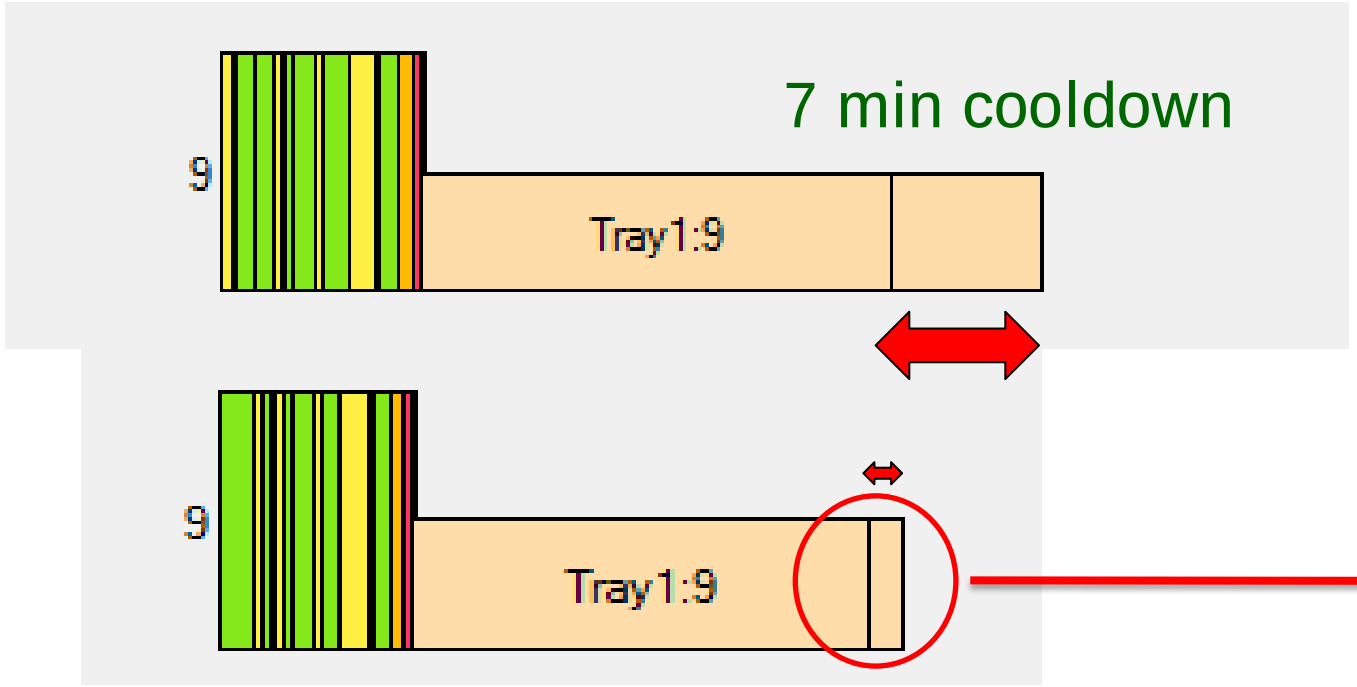


60 Samples per day – one instrument

10 samples with PrepAhead

Estimated Duration – 3 hours 59.51 mins

Phenols - CoolR^{PLUS}



13% of whole run time

Extra 9 samples a day

Overview of methods

Manual Methods

OCPs

1000mL Water/Sample
Add Cal/QC Standard
Mix
Weigh filled bottle (and empty)
If particulates present - dilute
Add Istd
Shake gently to mix
Add approx 20g salt
Add extraction solvent
Gently Shake
Release pressure (slack cap)
Shake 15-20mins (20max)
Release pressure (slack cap)
Add water to move solvent layer
Transfer ALL solvent layer to fresh tube
Centrifuge 1900rpm 10mins
Transfer ALL solvent layer to fresh tube
Evaporate to 1mL
Add salt to remove water
Inject 30µL

PAHs

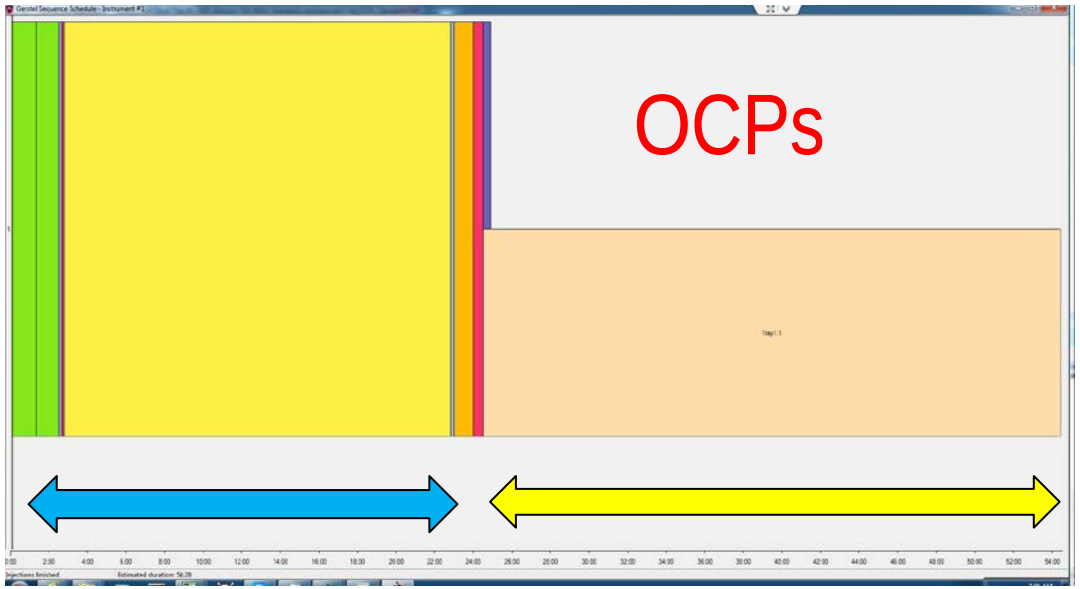
1000mL Water/sample
Add Cal/QC standard
Mix
Weigh sample
Visually inspect and dilute >= 1in2 if required
Add Istd
Shake gently to mix
Add extraction solvent
Shake (orbital shaker) 60mins 120rpm
Add salt
Add water to move solvent layer up
Remove emulsions
Centrifuge 2400rpm 10mins if required (emulsions)
Transfer cyclohexane to 10mL vial
Transfer 300uL to a microvial
Inject 10µL

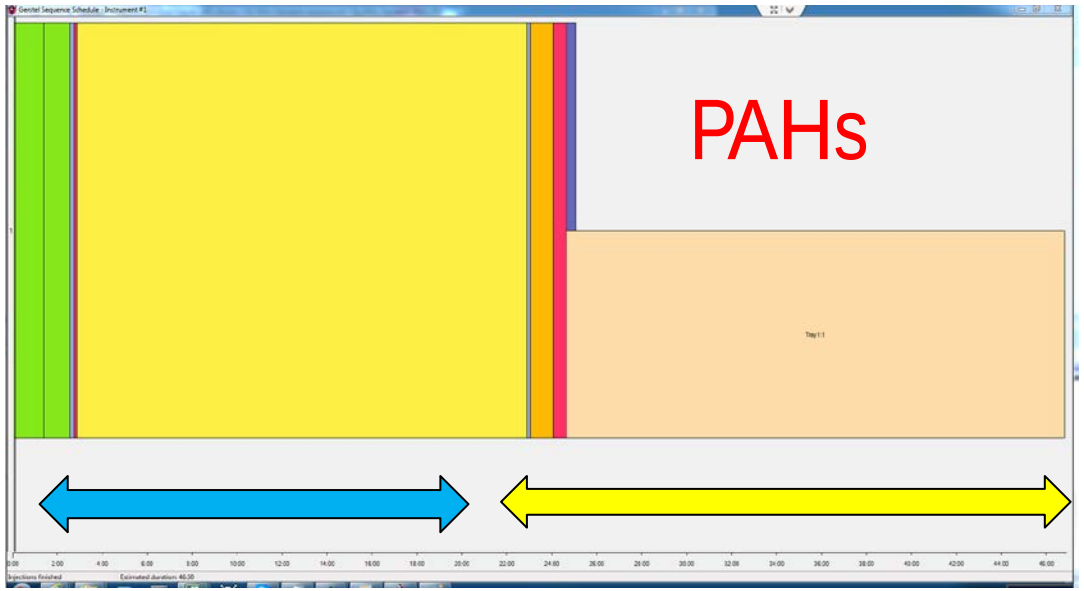
Overview of methods

Automated Methods

OCPs & PAHs
18mL Water
Spike Cals and QCs & Surrogate
Agitate 750rpm
Add 0.5mL extraction solvent
Agitate (15 or 60min)
Add 10µL Organic Solvent
Inject 10µL from solvent layer

Automation of OCP & PAH Sample Preparation





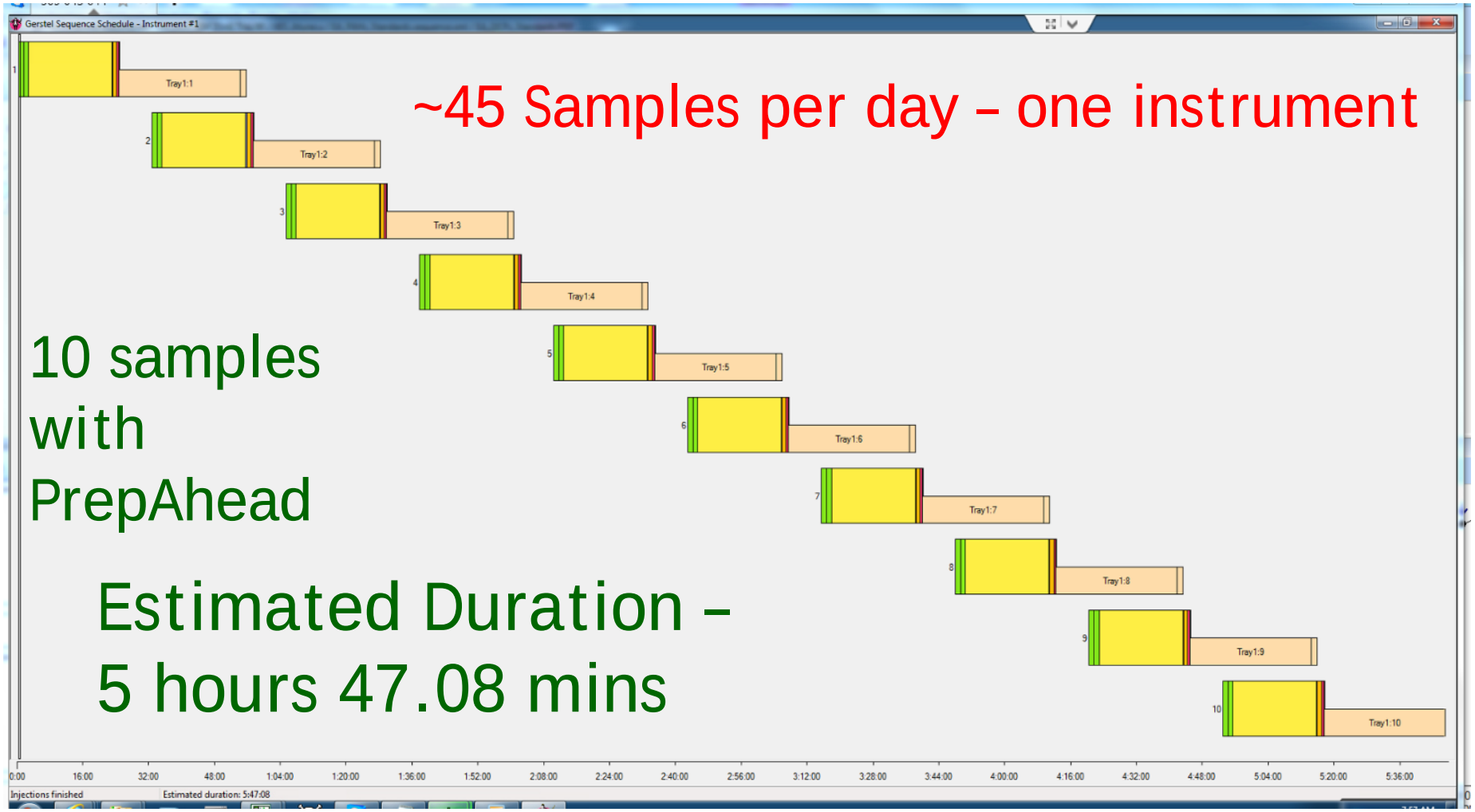
Sample Prep (22.58 mins) GC Run time (30 mins)

Estimated Duration ~ 53 mins

Sample Prep (24.19 mins) GC Run time (22.25 mins)

Estimated Duration ~ 47 mins

OCPs - PrepAhead

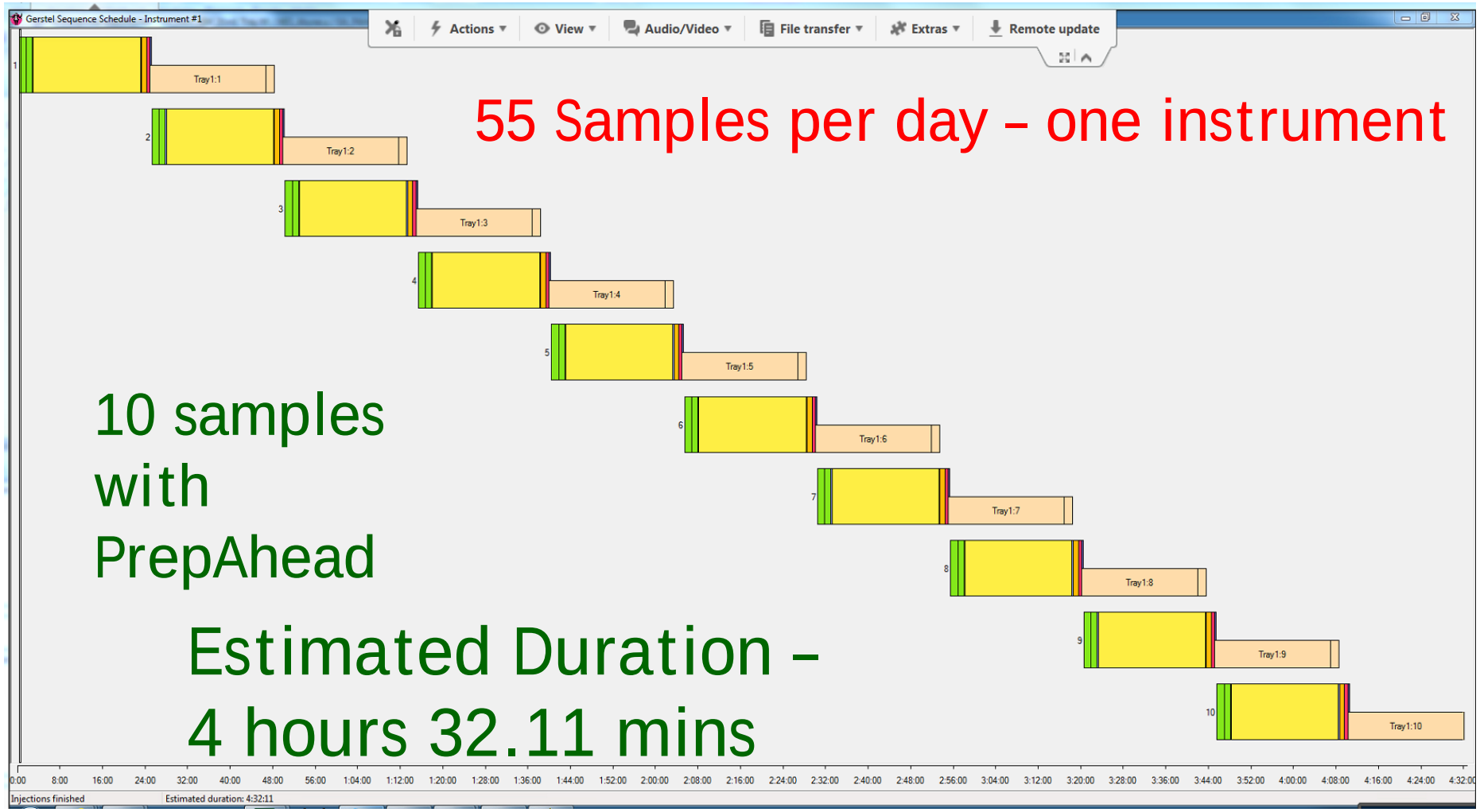


~45 Samples per day – one instrument

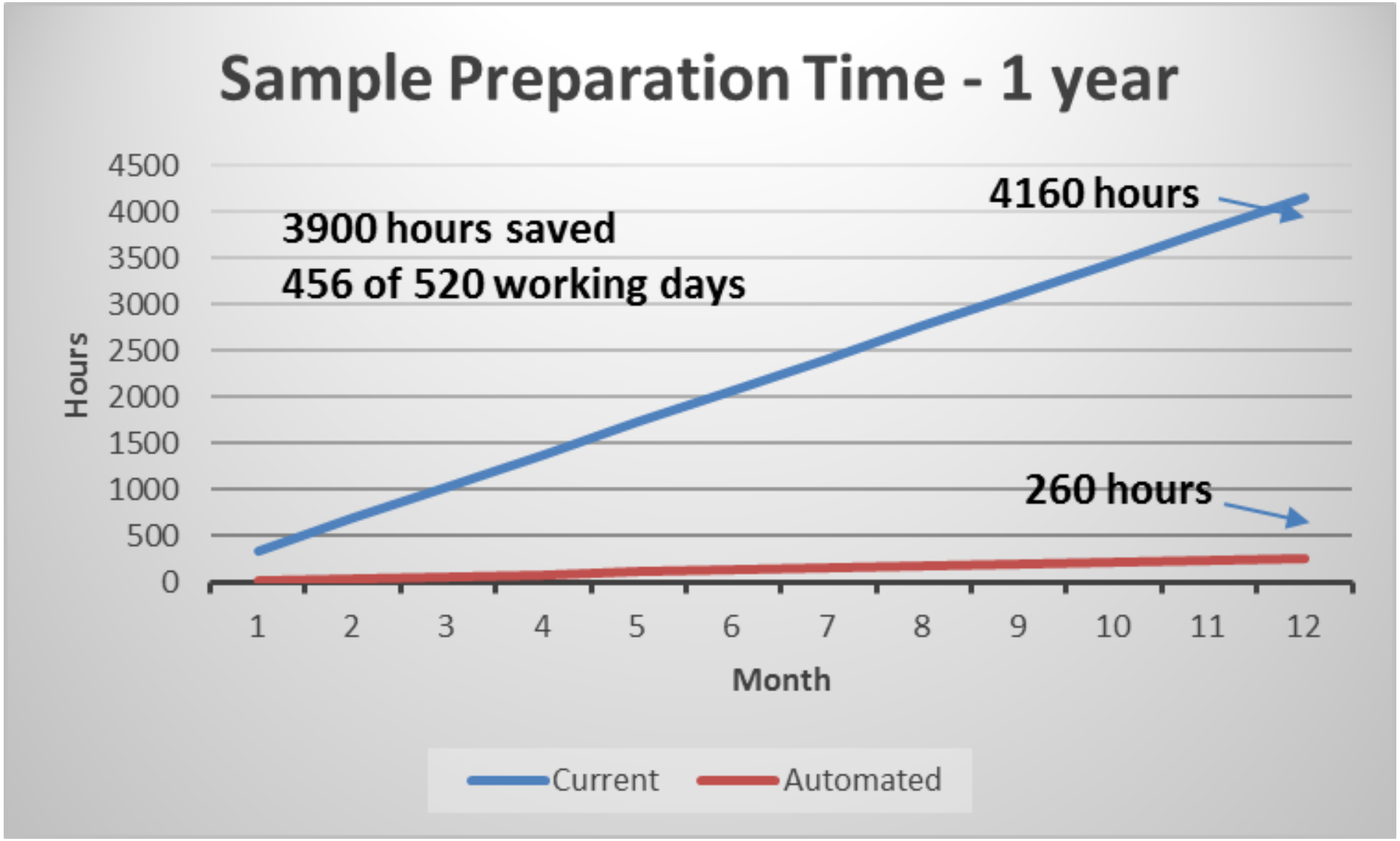
10 samples with PrepAhead

Estimated Duration – 5 hours 47.08 mins

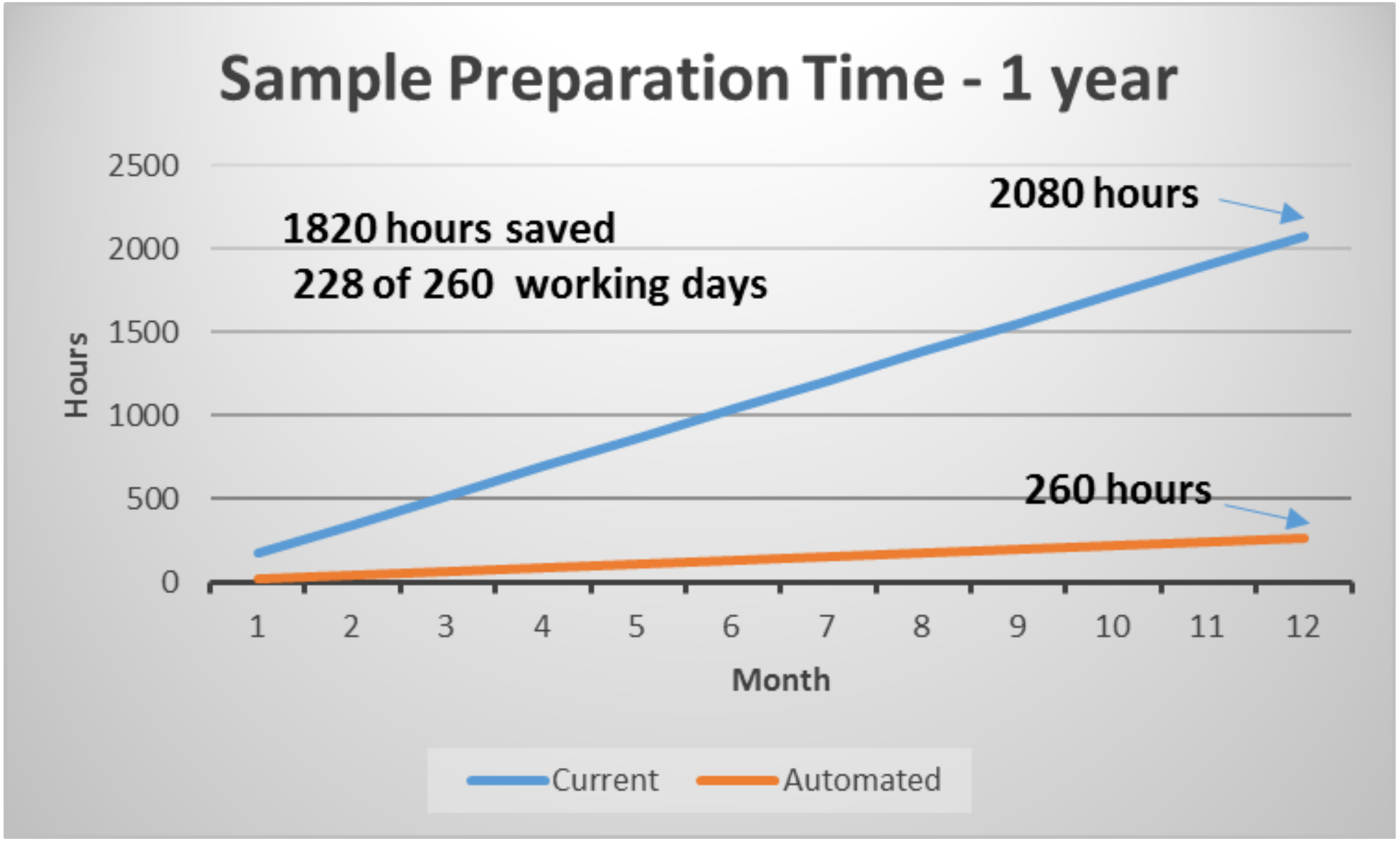
PAHs - PrepAhead



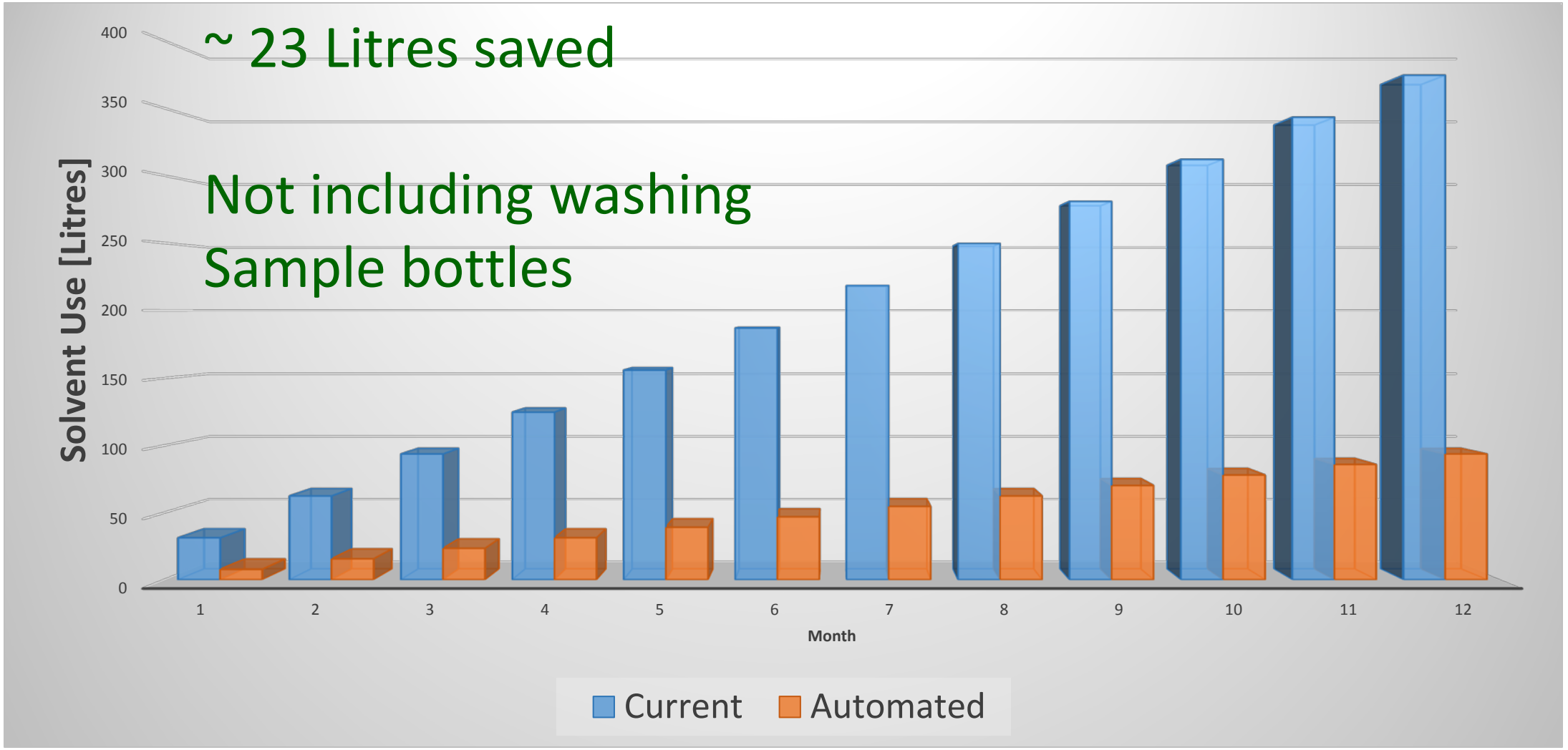
OCPs - PrepAhead



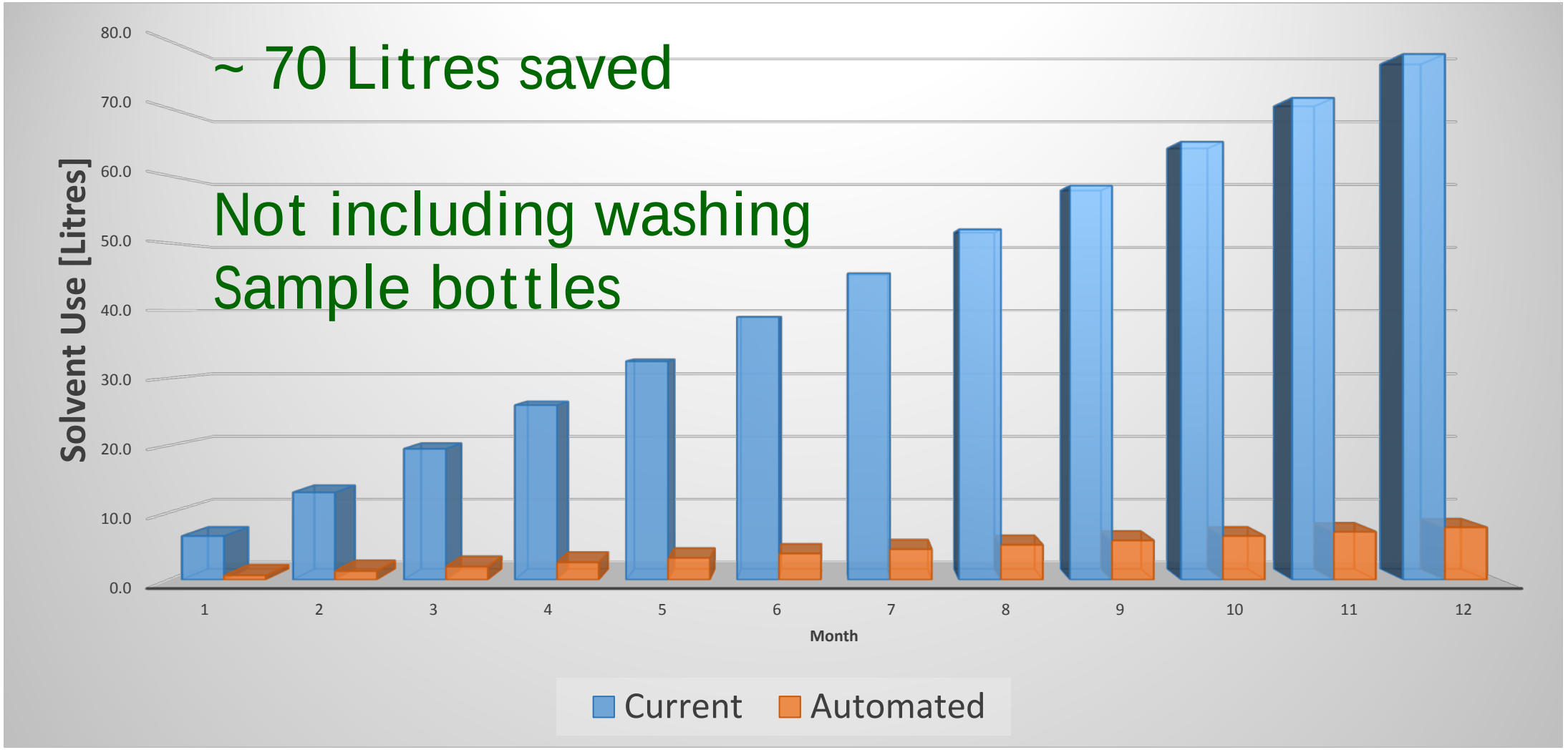
PAHs - PrepAhead



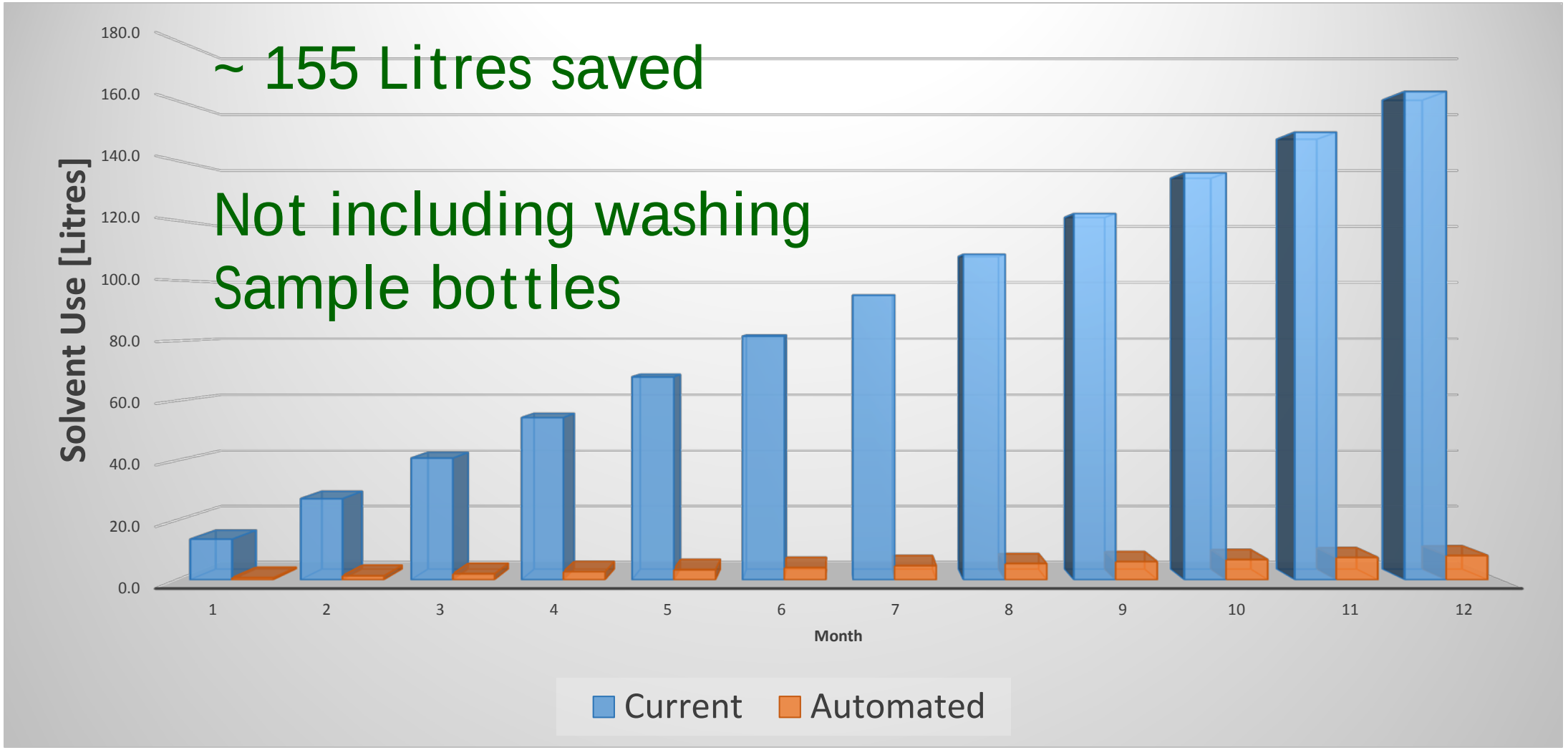
Phenols Solvent Consumption



PAHs Solvent Consumption



OCPs Solvent Consumption

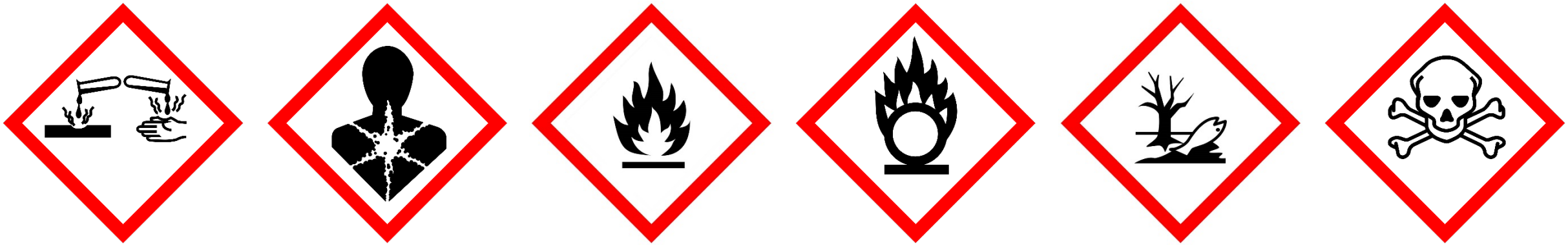


Solvent Consumption

Procedure	Solvent	Samples per day	Samples per year	Solvent use [Ltr] (manual)	Solvent use [Ltr] (automated)	Solvent saving	Solvent Cost (per Ltr)	Total Saving
Phenols	Solvent 1	60	15600	31.2	7.8	23.4		
PAHs	Solvent 2	60	15600	78	7.8	70.2		
OCPs	Solvent 3	66	17160	163	8.2	154.9		

Total Solvent reduction 272 Ltr

Total saving

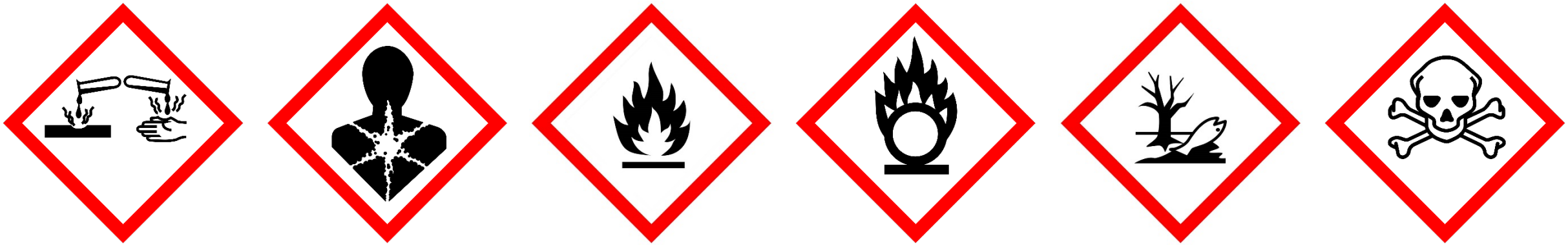


Solvent Consumption

Procedure	Solvent	Samples per day	Samples per year	Solvent use [Ltr] (manual)	Solvent use [Ltr] (automated)	Solvent saving	Solvent Cost (per Ltr)	Total Saving
Phenols	Solvent 1	60	15600	31.2	7.8	23.4	£25	£585
PAHs	Solvent 2	60	15600	78	7.8	70.2	£25	£1755
OCPs	Solvent 3	66	17160	163	8.2	154.9	£25	£3872

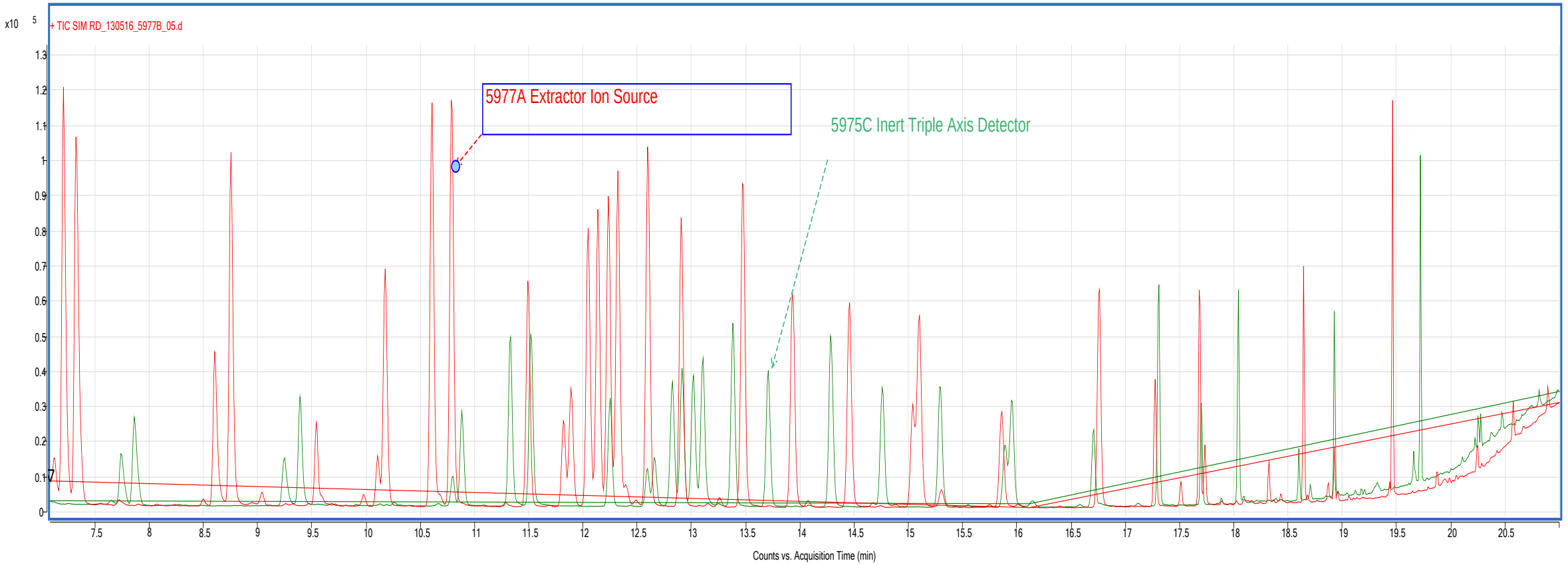
Total Solvent reduction 272 Ltr

Total saving £6212



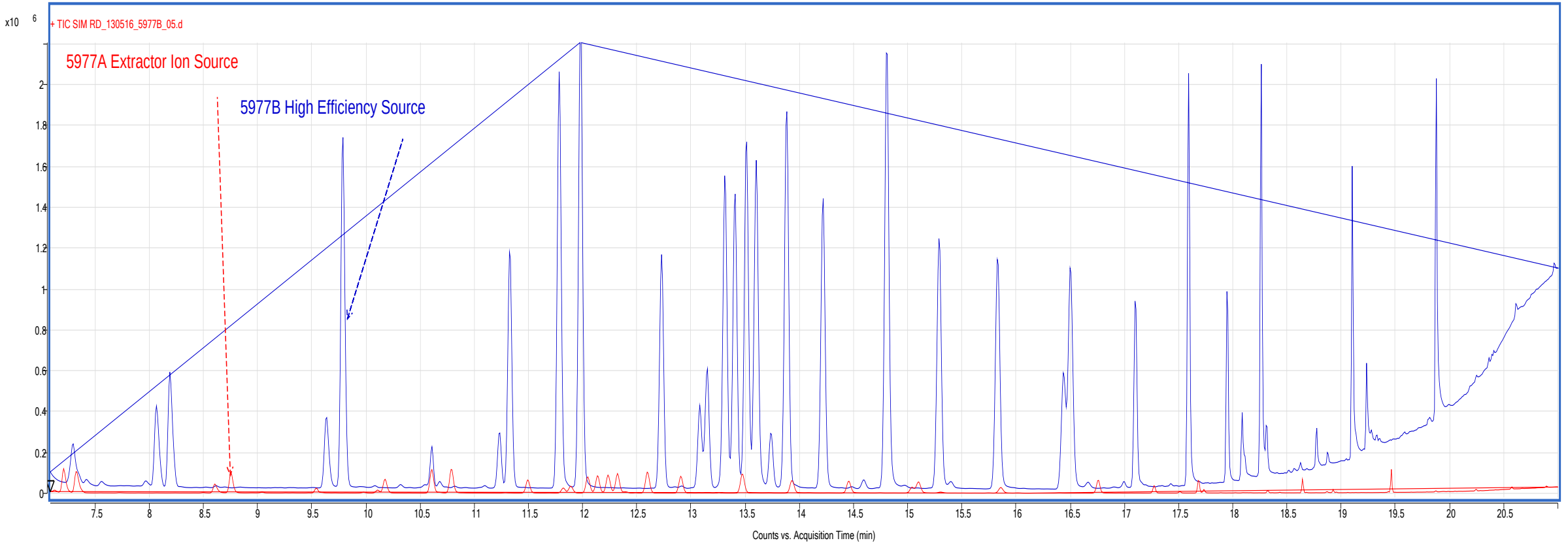
High Efficiency Source - Sensitivity

Overlaid chromatograph of 5975C and 5977A

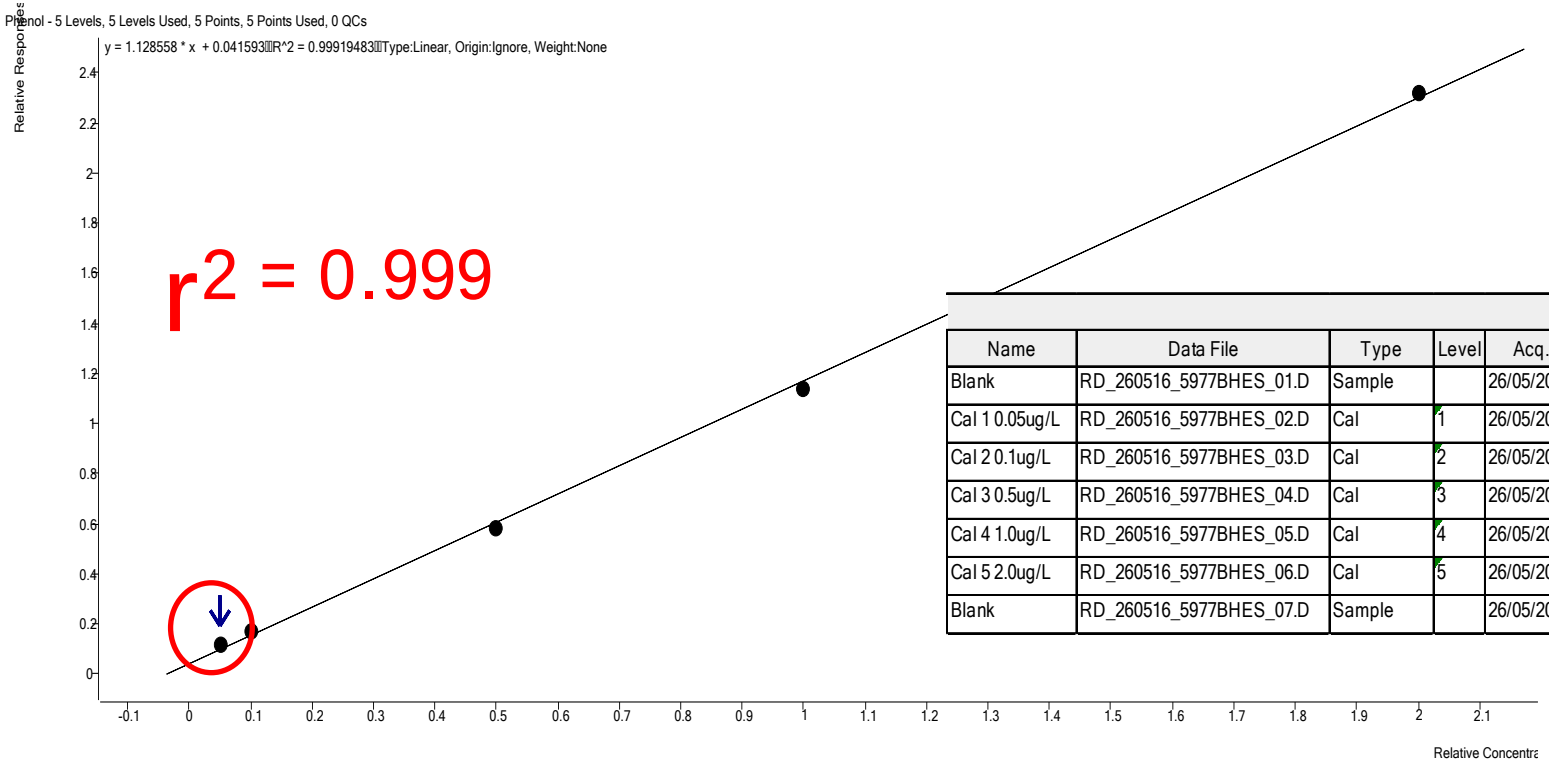


High Efficiency Source - Sensitivity

Overlaid chromatograph of 5977B HES vs 5977A Extractor Ion Source

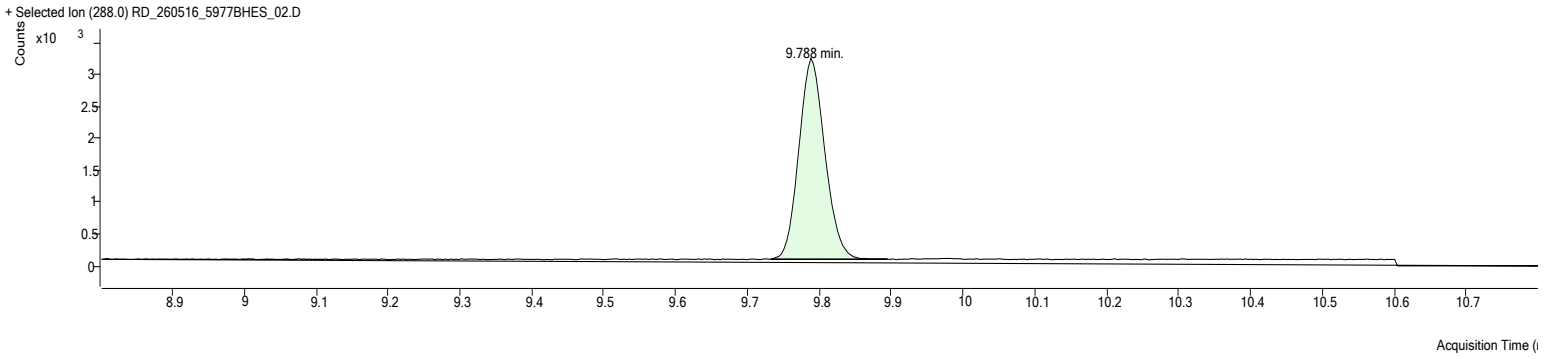


Linearity and Precision - Phenols



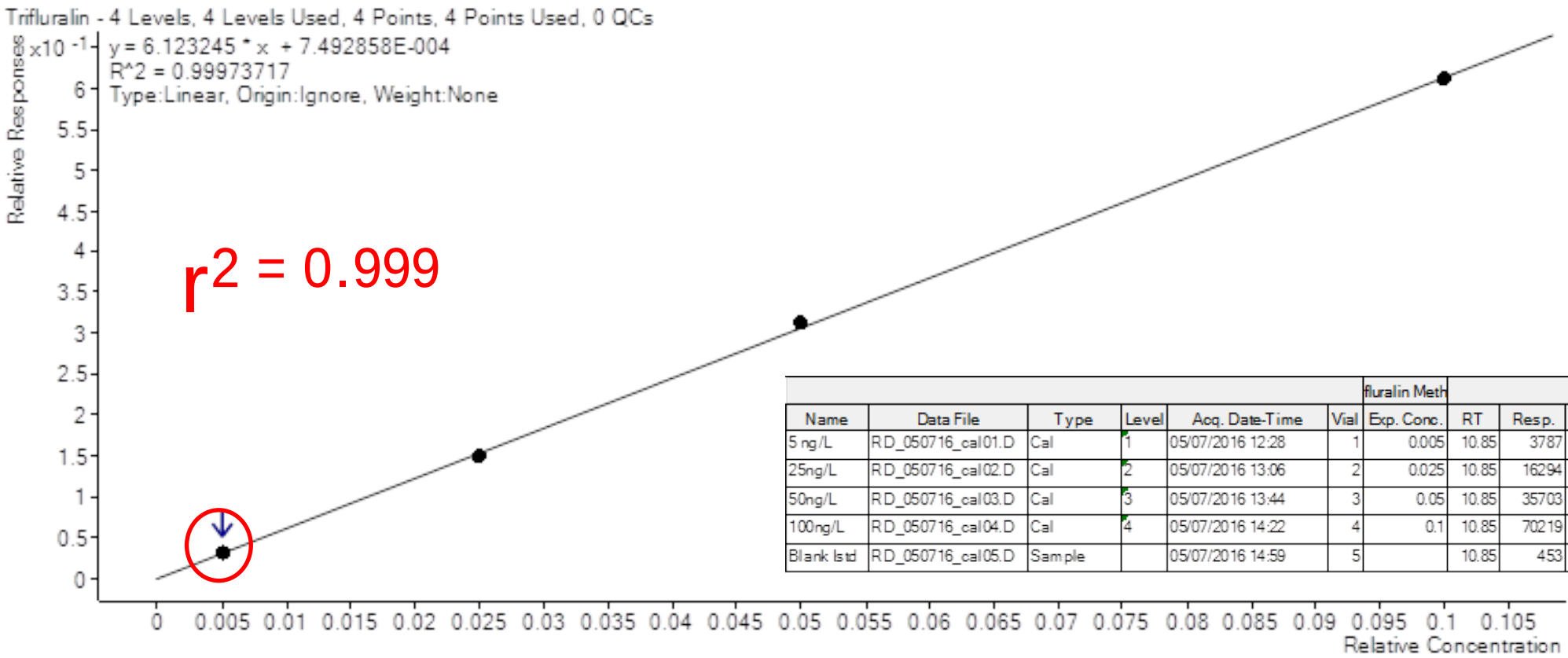
$r^2 = 0.999$

					Phenol Metho	Phenol Results					Phenol-d6 (ISTD) Results	
Name	Data File	Type	Level	Acq. Date-Time	Exp. Conc.	RT	Resp.	Calc. Conc.	Final Conc.	Accuracy	RT	Resp.
Blank	RD_260516_5977BHES_01.D	Sample		26/05/2016 16:47		9.79	4299.1	0.0059	0.0059		9.75	65128
Cal 1 0.05ug/L	RD_260516_5977BHES_02.D	Cal	1	26/05/2016 17:14	0.05	9.79	7926.3	0.0519	0.0519	103.7	9.75	67867
Cal 2 0.1ug/L	RD_260516_5977BHES_03.D	Cal	2	26/05/2016 17:41	0.1	9.79	12106.3	0.1020	0.1020	102.0	9.74	70329
Cal 3 0.5ug/L	RD_260516_5977BHES_04.D	Cal	3	26/05/2016 18:09	0.5	9.79	41188.5	0.4701	0.4701	94.0	9.75	71201
Cal 4 1.0ug/L	RD_260516_5977BHES_05.D	Cal	4	26/05/2016 18:36	1	9.79	78081.2	0.9796	0.9796	98.0	9.75	68431
Cal 5 2.0ug/L	RD_260516_5977BHES_06.D	Cal	5	26/05/2016 19:03	2	9.79	168785.2	2.0465	2.0465	102.3	9.75	72791
Blank	RD_260516_5977BHES_07.D	Sample		26/05/2016 19:30		9.79	4517.6	0.0063	0.0063		9.75	67945



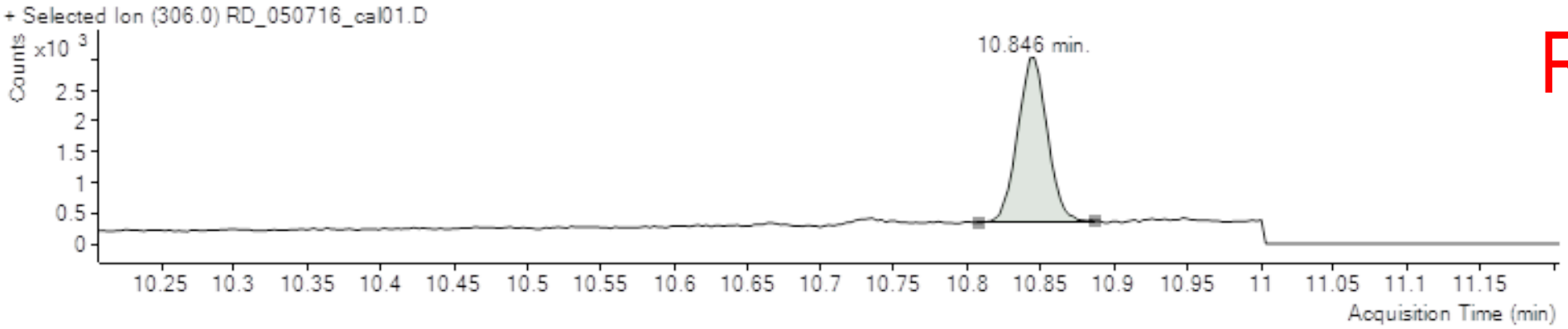
RSD= 3.7%

Linearity and Precision - OCPs



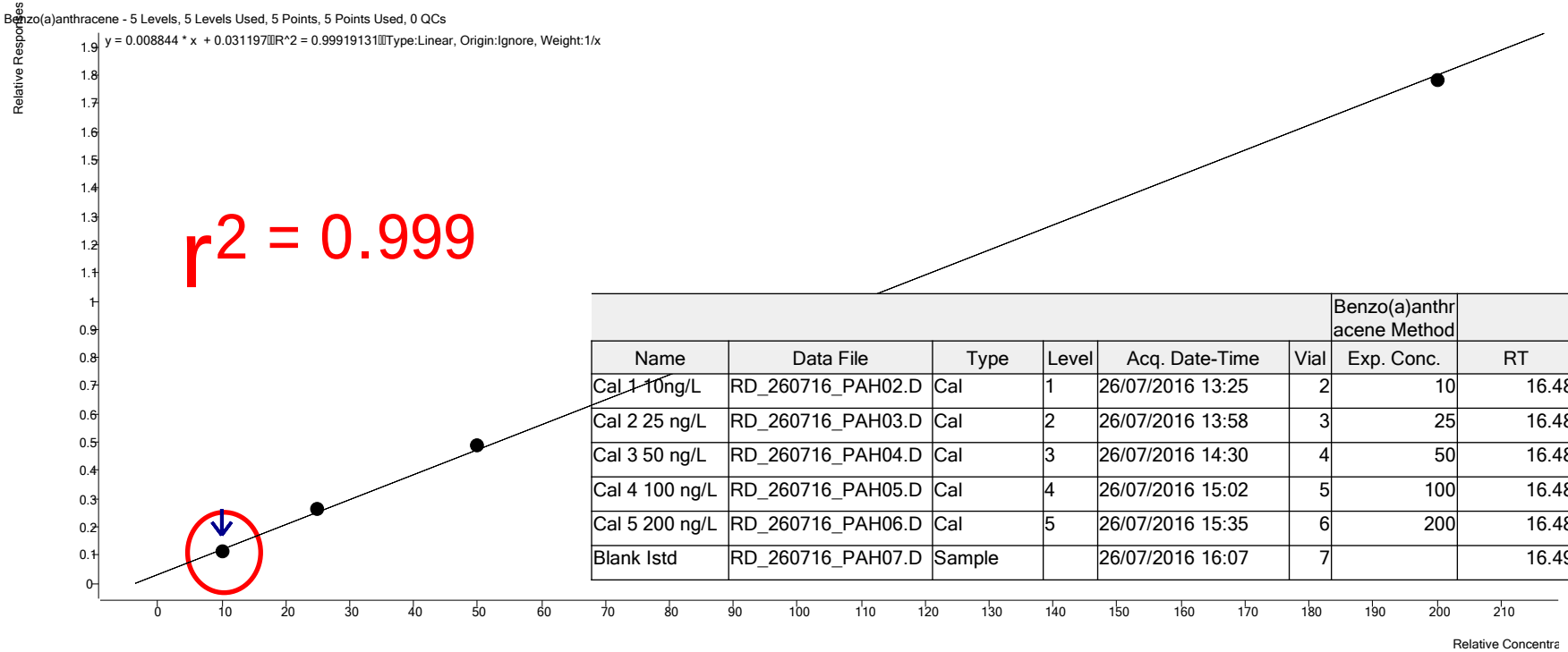
$r^2 = 0.999$

						fluralin Meth	Trifluralin Results					alin-d14 (IST D) R	
Name	Data File	Type	Level	Acq. Date-Time	Vial	Exp. Conc.	RT	Resp.	Calc. Conc.	Final Conc.	Accuracy	RT	Resp.
5 ng/L	RD_050716_cal01.D	Cal	1	05/07/2016 12:28	1	0.005	10.85	3787	0.0051	0.0051	102.2	10.74	118135
25ng/L	RD_050716_cal02.D	Cal	2	05/07/2016 13:06	2	0.025	10.85	16294	0.0243	0.0243	97.1	10.74	109024
50ng/L	RD_050716_cal03.D	Cal	3	05/07/2016 13:44	3	0.05	10.85	35703	0.0509	0.0509	101.7	10.74	114369
100ng/L	RD_050716_cal04.D	Cal	4	05/07/2016 14:22	4	0.1	10.85	70219	0.0997	0.0997	99.7	10.74	114830
Blank Istd	RD_050716_cal05.D	Sample		05/07/2016 14:59	5		10.85	453	0.0006	0.0006		10.74	102474



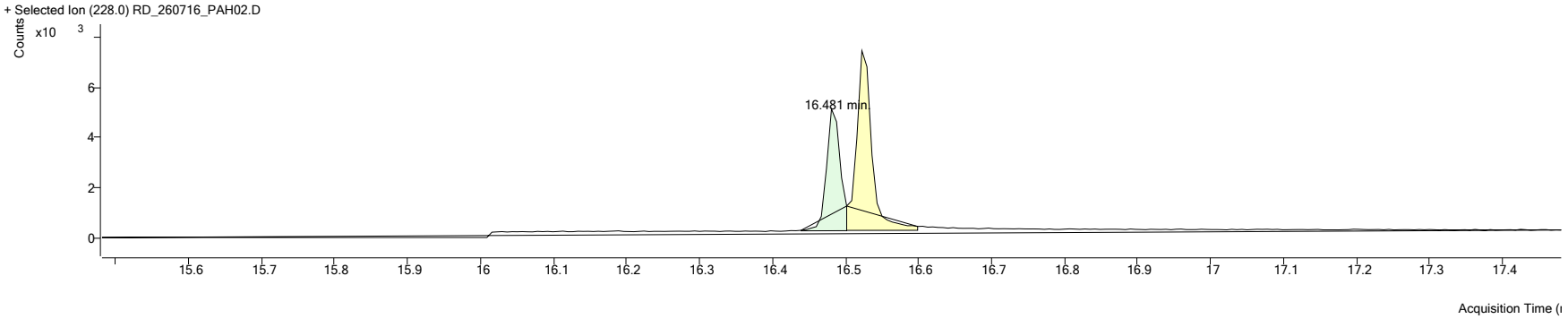
RSD= 5.5%

Linearity and Precision - PAHs



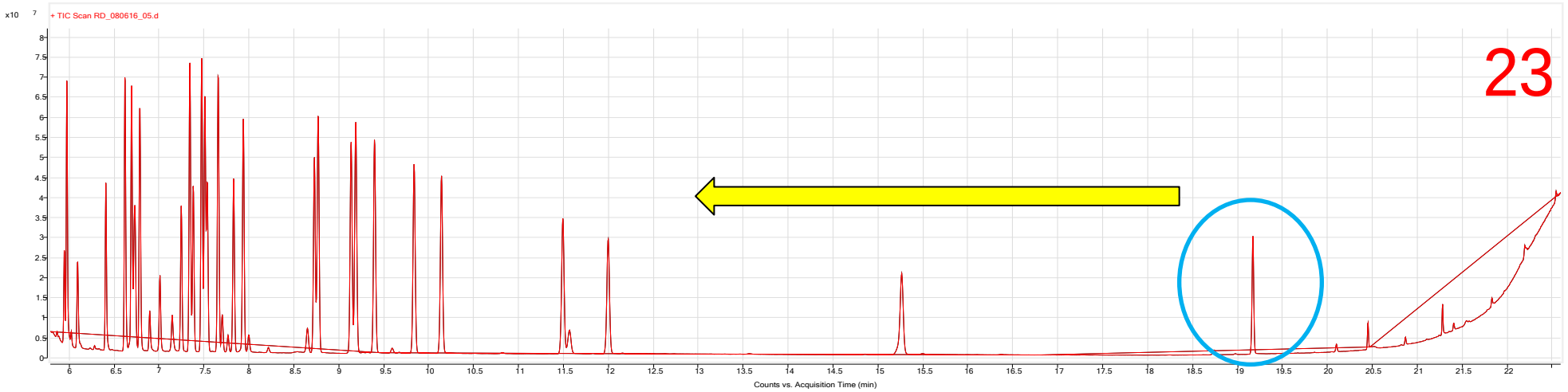
$r^2 = 0.999$

						Benzo(a)anthracene Method	Benzo(a)anthracene Results					Benzo(a)Anthracene-d12 (ISTD) Results	
Name	Data File	Type	Level	Acq. Date-Time	Vial	Exp. Conc.	RT	Resp.	Calc. Conc.	Final Conc.	Accuracy	RT	Resp.
Cal 1 10ng/L	RD_260716_PAH02.D	Cal	1	26/07/2016 13:25	2	10	16.48	6346	9.3	9.3	92.6	16	56110
Cal 2 25 ng/L	RD_260716_PAH03.D	Cal	2	26/07/2016 13:58	3	25	16.48	15229	26.4	26.4	105.6	16	57517
Cal 3 50 ng/L	RD_260716_PAH04.D	Cal	3	26/07/2016 14:30	4	50	16.48	32573	51.5	51.5	103.0	16	66960
Cal 4 100 ng/L	RD_260716_PAH05.D	Cal	4	26/07/2016 15:02	5	100	16.48	51629	99.7	99.7	99.7	16	56535
Cal 5 200 ng/L	RD_260716_PAH06.D	Cal	5	26/07/2016 15:35	6	200	16.48	111117	198.1	198.1	99.1	16	62307
Blank Istd	RD_260716_PAH07.D	Sample		26/07/2016 16:07	7		16.49	953	0.0	0.0		16	57722

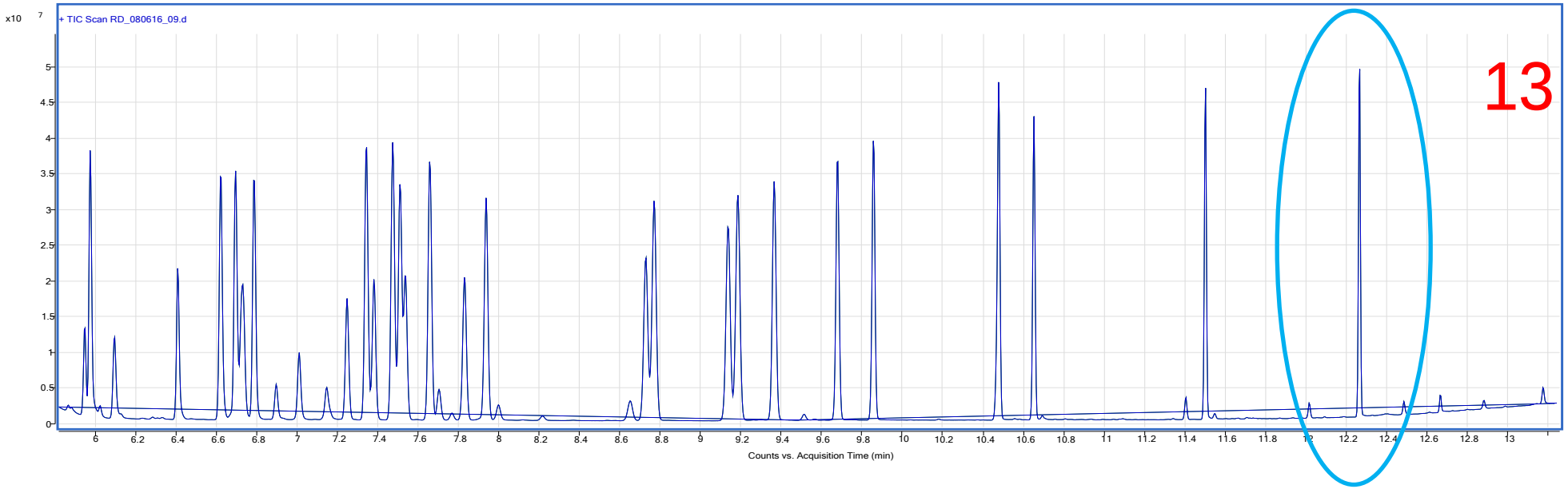


RSD= 7%

BSTFA runtime



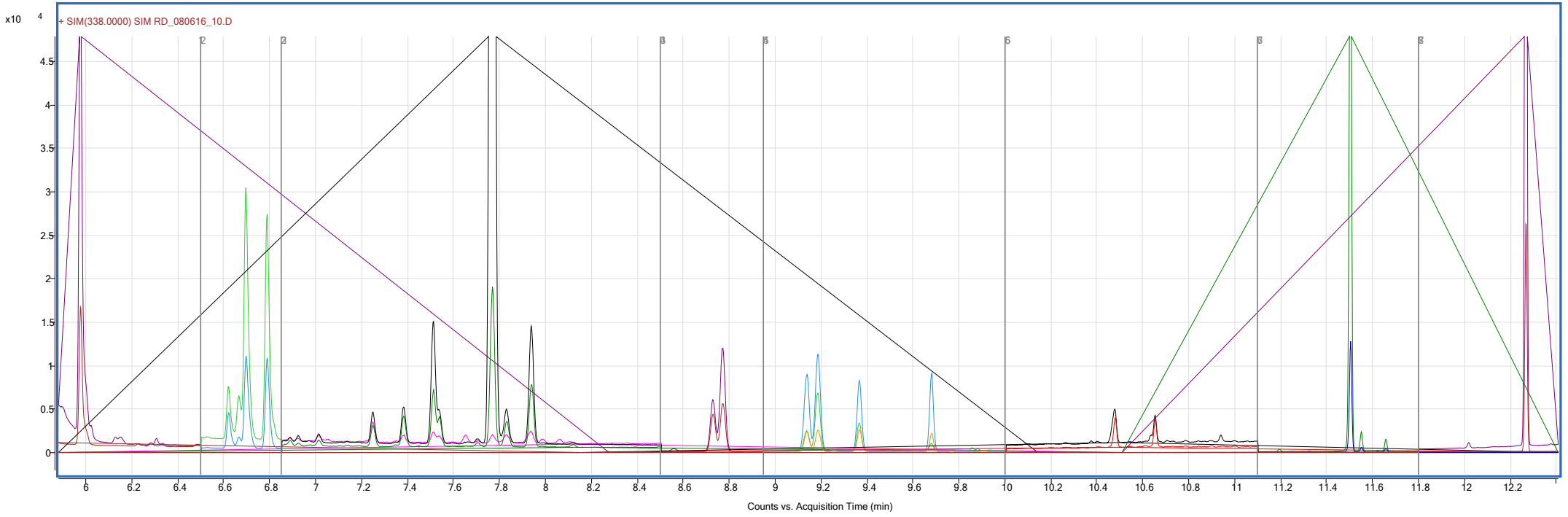
23 mins



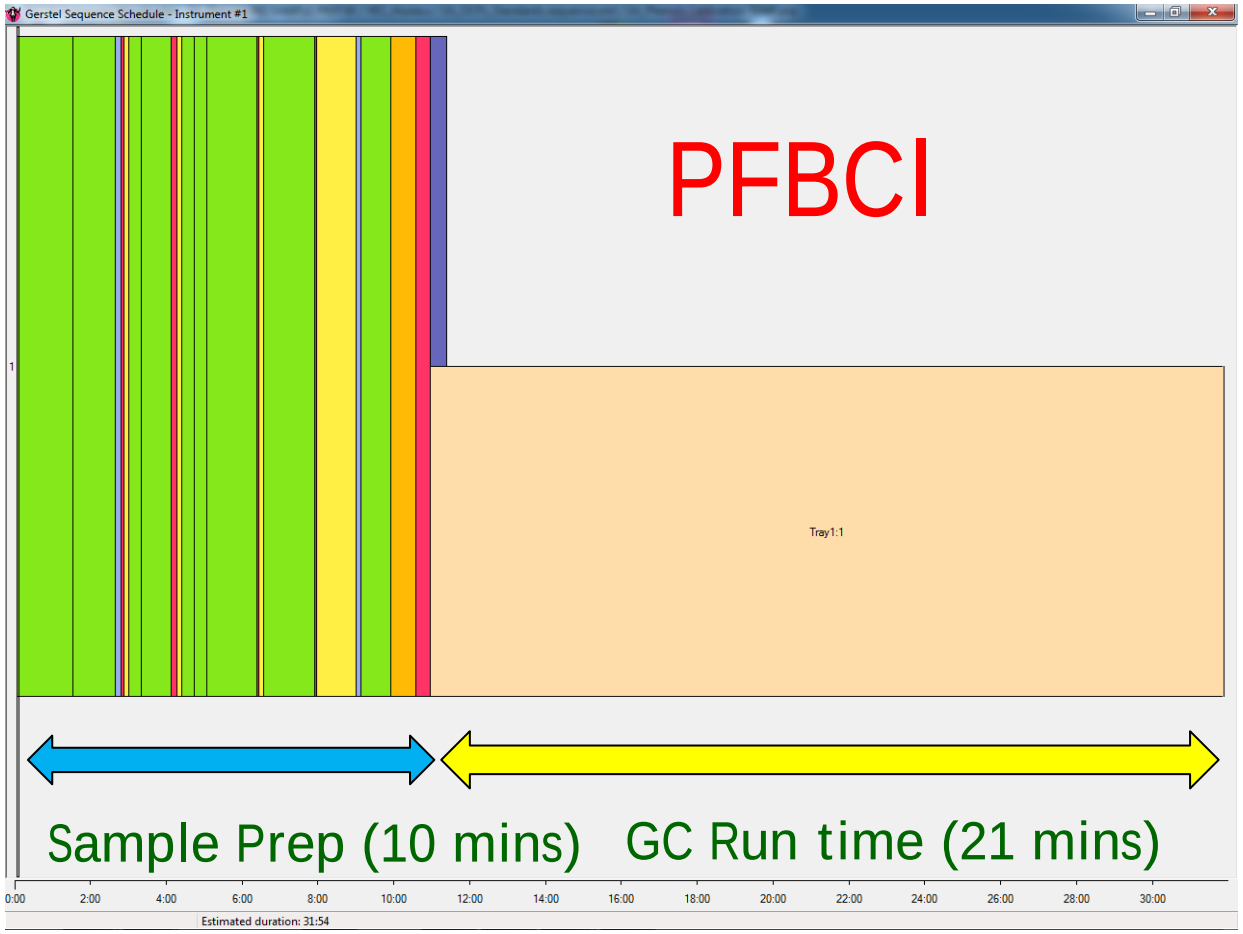
13 mins

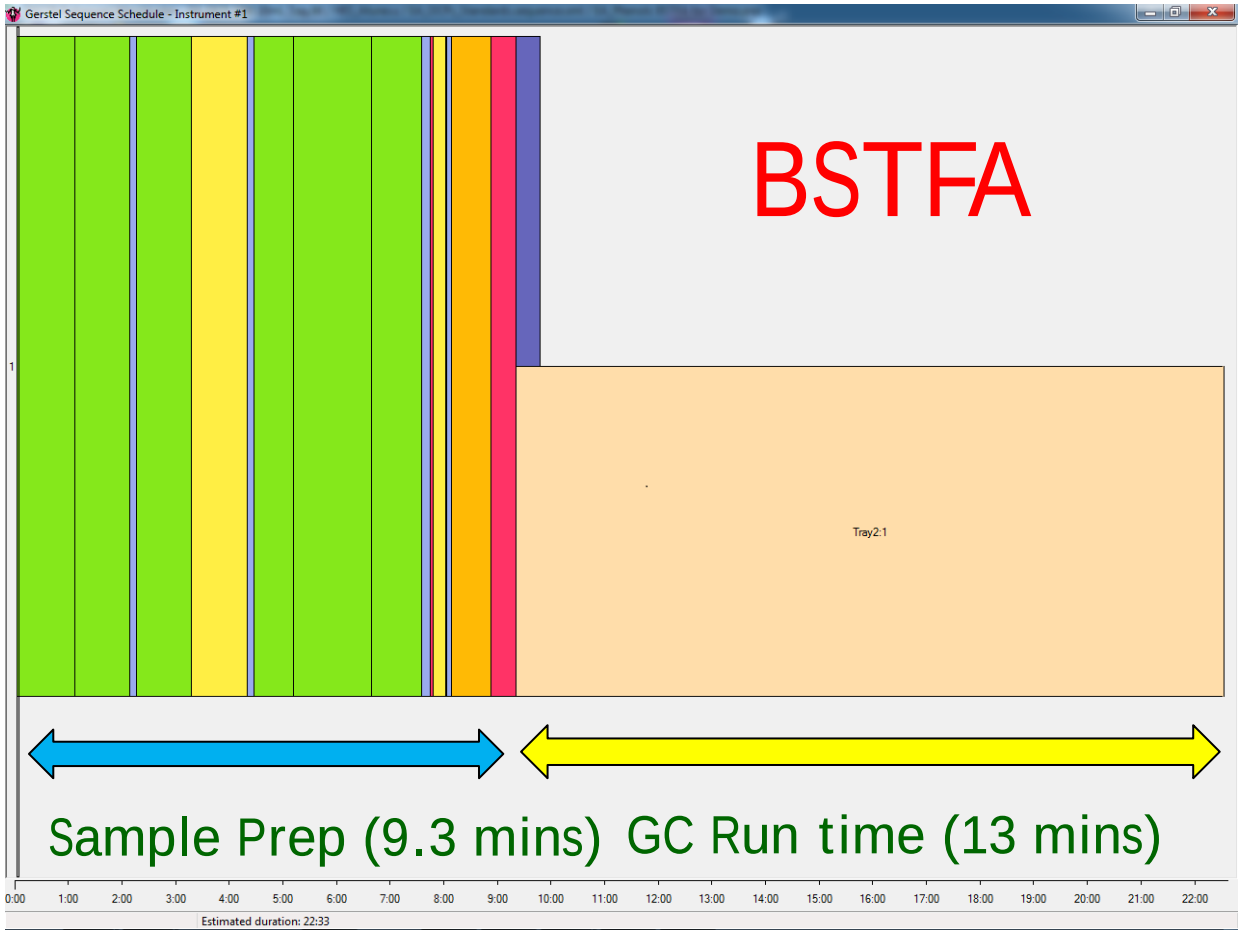
BSTFA

Phenols - SIM ions at 0.05 µg/L
1.0µL injected into split/splitless inlet



Automation of Sample Preparation

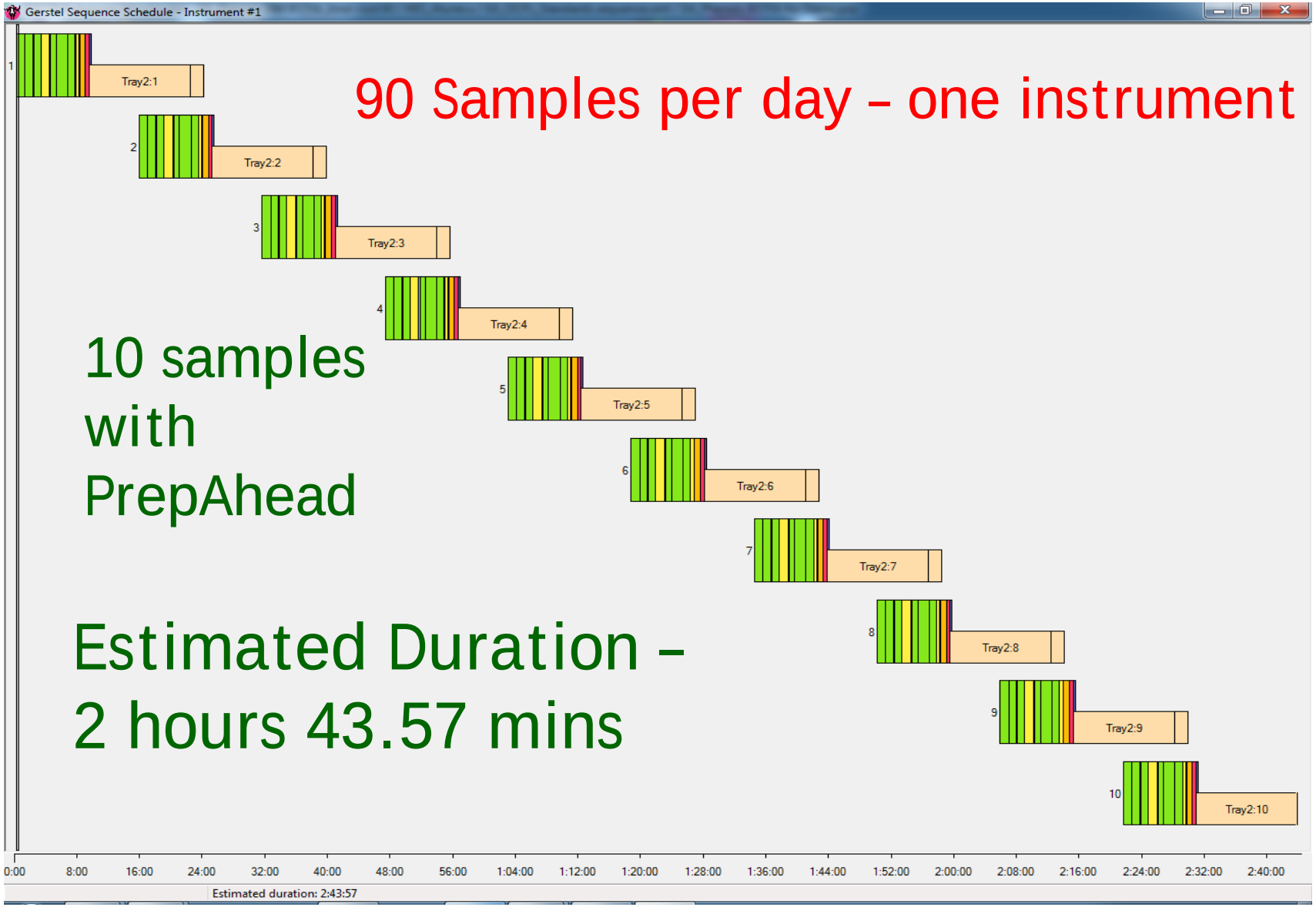




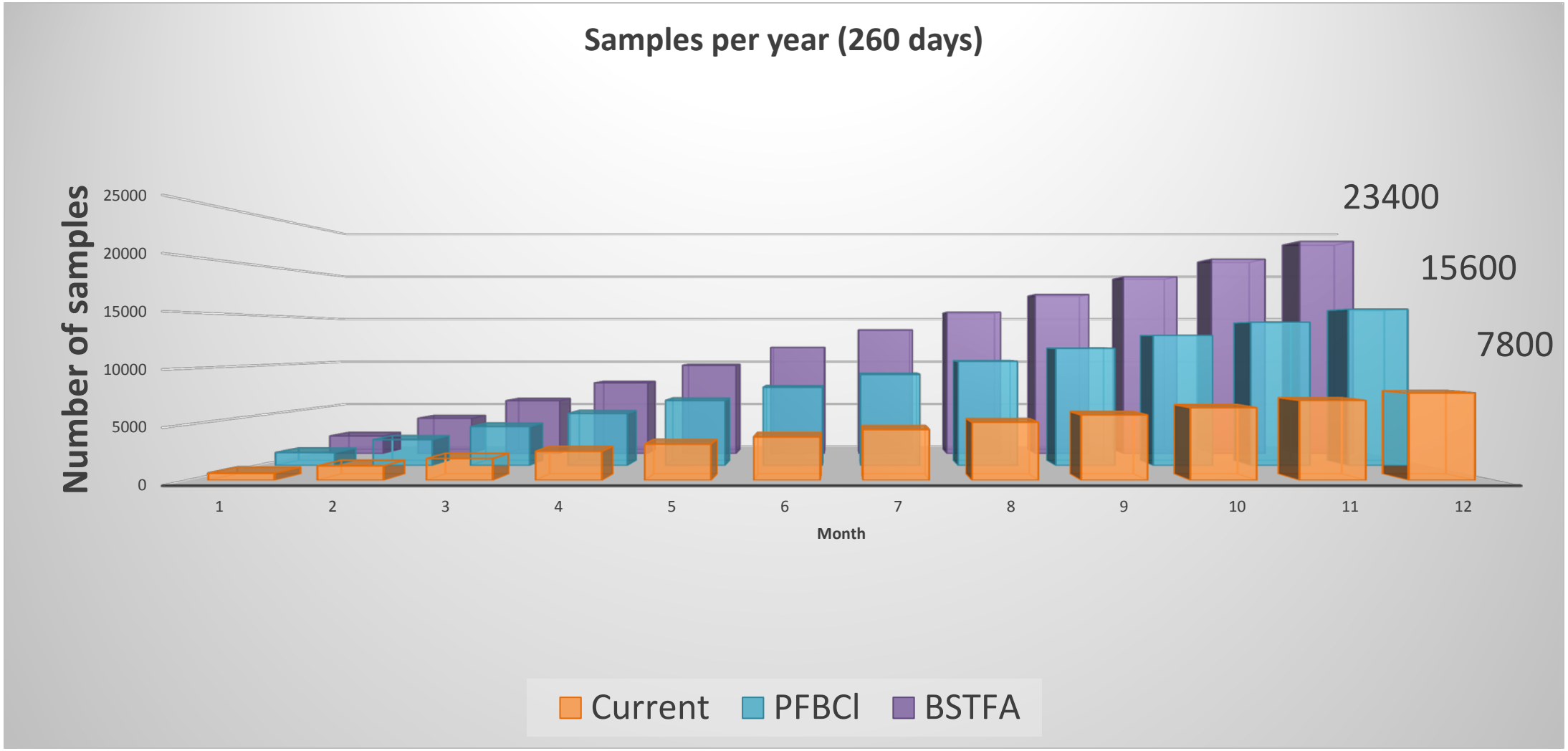
Estimated Duration – 31.54 mins

Estimated Duration – 22.33 mins

Automation of Sample Preparation



Comparison of Phenol Methods



Summary

- Sample throughput
- Turnaround times
- Analyst time
- Accuracy and precision
- Storage
- Disposal
- Transport
- Safety

Summary

- Small Injection size
  - Less frequent maintenance
  - Better Data Quality
  - Scaled down method
  - Costs of solvents
- Less sample prep – analyst time
- Smaller Lab Footprint
- Lower detection limits