Challenges Seen in the Development and Delivery of EPA 537 R1.1 (and DOD-modification)
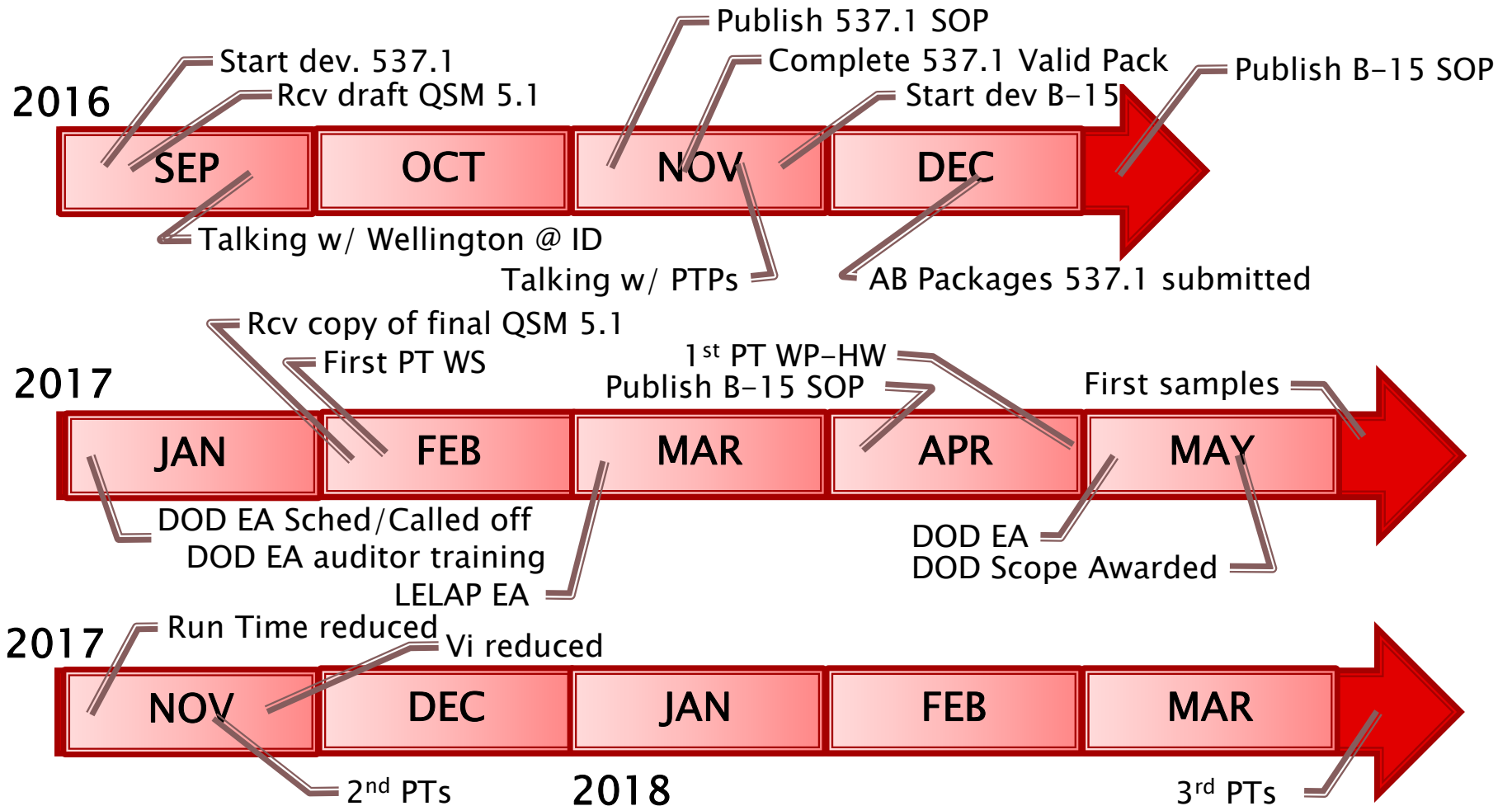
August 9, 2018

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Summer of 2016 this was sitting on a bench not yet running – waiting for the market to develop.

GCAL ANALYTICAL LABORATORIES, LLC

Development/Certification

2016

Publish 537.1 SOP
Start dev. 537.1
Rcv draft QSM 5.1
Complete 537.1 Valid Pack
Publish B-15 SOP
Start dev B-15

| SEP | OCT | NOV | DEC |

Talking w/ Wellington @ ID
Talking w/ PTPs
AB Packages 537.1 submitted

2017

Rcv copy of final QSM 5.1
First PT WS
1st PT WP-HW
Publish B-15 SOP
First samples

| JAN | FEB | MAR | APR | MAY |

DOD EA Sched/Called off
DOD EA auditor training
LELAP EA
DOD EA
DOD Scope Awarded

2017

Run Time reduced
Vi reduced

| NOV | DEC | JAN | FEB | MAR |

2nd PTs
2018
3rd PTs

2018 also:
- AF 3x blind study (March)
- Client EA (July – focused on PFAS)
- DOD EA (July – 2x reviewing PFAS)

Internal

- EIS mapping
- Standards sourcing
- MeOH contamination
- HPLC water batching
- Sample container specification
- Run duration change - Delay column impact - PFOS/PFHxS underestimation
- Decreasing required sample volume
- Cartridge contamination
- 3 PT targets fail (out of 360 targets tested – we do all targets)
- Validator training on how to read the trace report.
- Ion Ratio reset

External

- 1st DOD EA findings
- DOD validation package requests
- Level IV package needs
- Client EA Finding: variance with other labs (PFOS/HFHxS underestimation due to baseline not extending to branch-chained isomers)
- 2nd DOD EA findings.
- Blank contamination (or is it time to replace the delay column?) *NEW*

Issue	Lesson Learned	Client Impact
HPLC Water	Can use our own water but must have a batching/tracking system.	None
Teflon liners	Order MeOH that does not come with this, also sample containers, water containers.	Interferences
Delay column	Don't assume: delays contaminants, not analytes. Run natives whenever changing method to assure all isomers are located. These apparently have short life spans.	Bias low, Blank contamination.
Total PFOS/PFHxS	Assumption lead to baseline not being extended.	Bias low
Validator training	Total PFOS/PFHxS issue could easily been caught.	Bias low
Bad cartridge lot	Assumption that cartridges remain of equal quality from batch to batch.	Bias high
Ion Ratio reset	QC controls are only good if they are understood, managed properly and used	False negatives

GCAL Performance with PFOA in waters
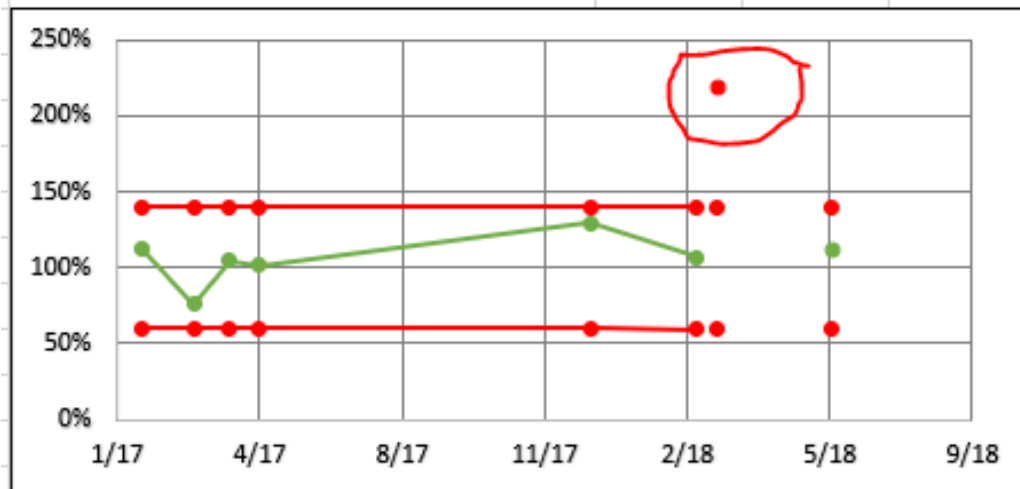
GCAL Performance with PFOS in waters
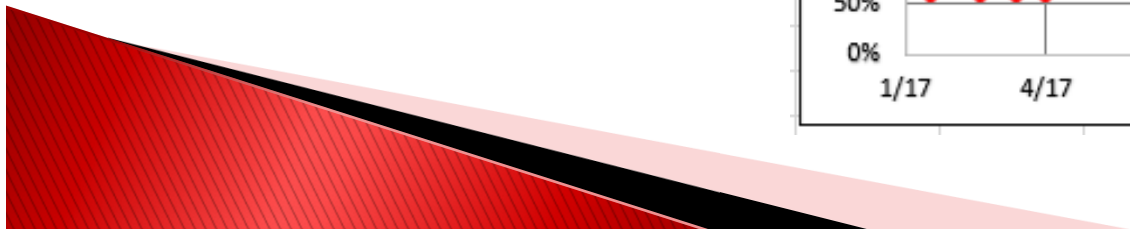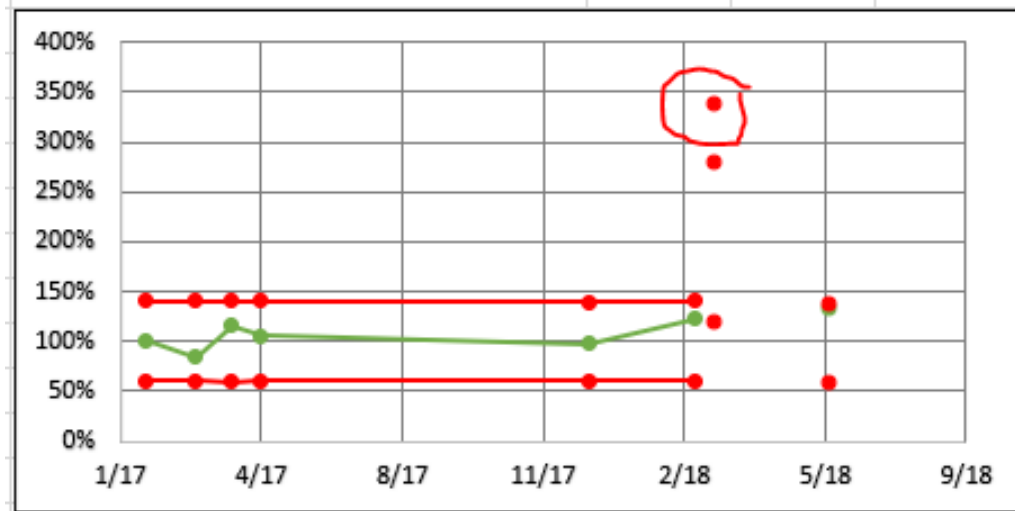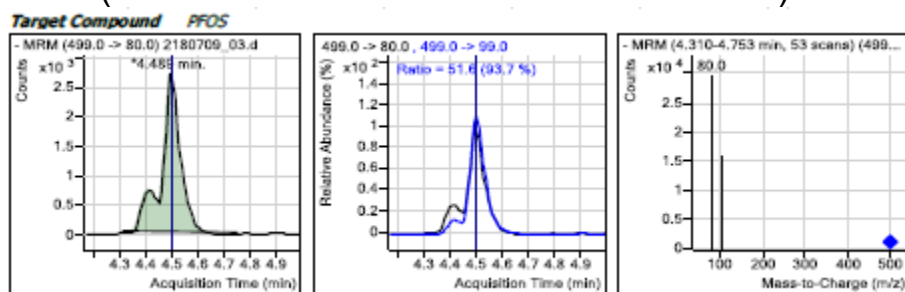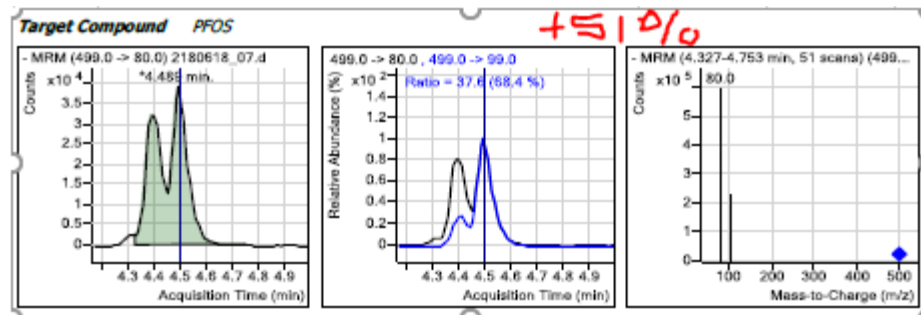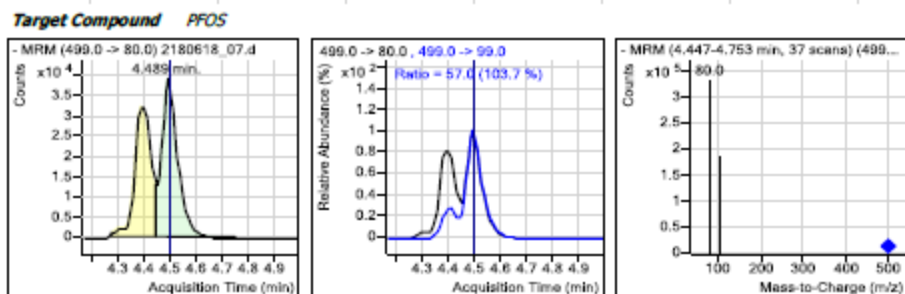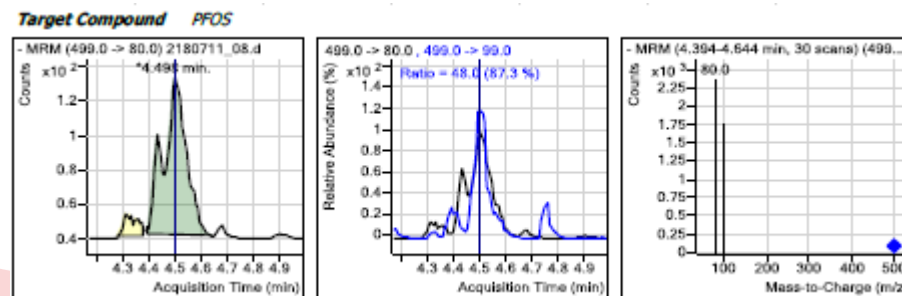
Significance (PFOS):

~10% (with minimal branch-chained enrichment)

~50% (with significant branch-chained enrichment)
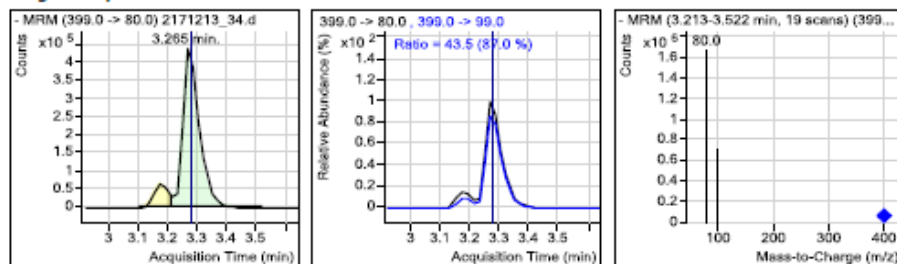

Note: %D can vary wildly at low concentrations ("J")

Significance (PFHxS):

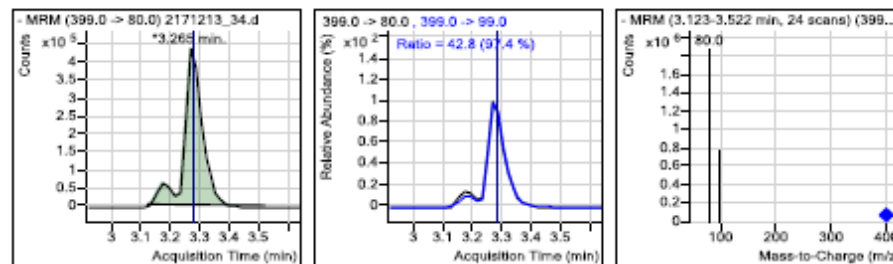0% (with minimal branch-chained enrichment)

~50% (with significant branch-chained enrichment)
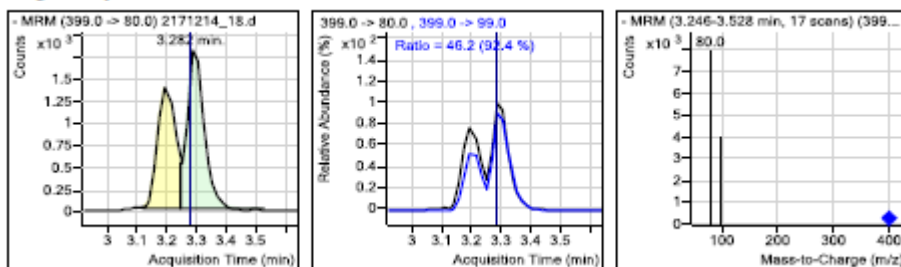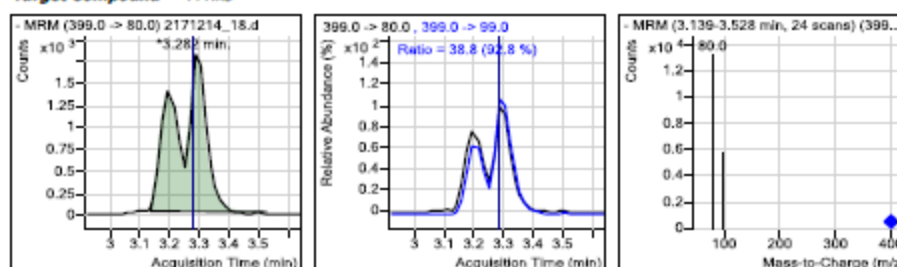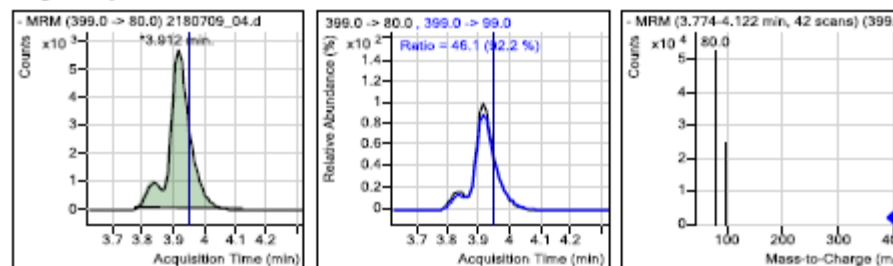

Note: %D can vary wildly at low concentrations ("J"). Also redrawing of baselines can cause redraw results to be less than original results.
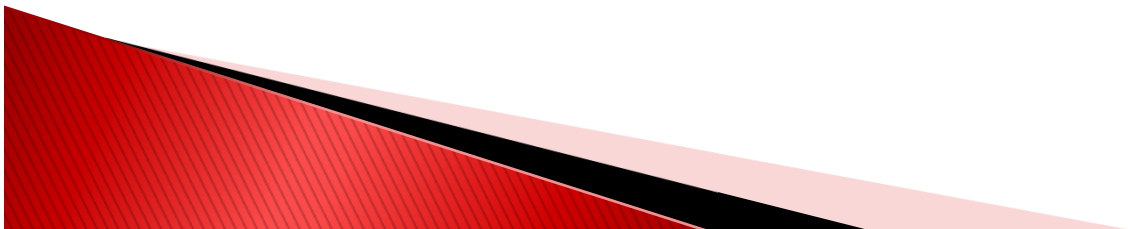
- A rough few years, a lot of questions, many jumps to assumptions by lab and clients, a complicated learning curve.
- We are only where we are because of the challenges thrown our way by competitors and customers.
- LOE by QAM: About 4 hours every 2 weeks since beginning responding to questions and issues.

For more information:

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