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Performance of Laboratories Undertaking the Determination of Benzene in Air, as Codified in EPA TO-17: Results from the International AIR PT Scheme

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Overview of presentation

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- Overview of the AIR PT scheme, the TO-17 test sample and how it is prepared
- Laboratory PT performance over 10 recent rounds
- Laboratory performance in relation to measurement quality objectives (MQOs: EU *v* US)
- Conclusions
- Future PT research/developments

AIR Proficiency Testing Scheme

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- April 2014 – Partnership between HSL and LGC PT
- Joins HSL WASP PT with LGC STACK PT schemes
  - PT samples for ambient, indoor, workplace and stack air testing
- HSL WASP PT started in 1988
  - 100 PT rounds completed by March 2014
  - EPA TO-17 sample type started in Round 35 (1997)

AIR PT sample details

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- Tenax TD tubes spiked with BTEX
- Spiked in the range 25 – 1000 ng
  - Most spikes at 100 ng or lower
  - Spike data in this presentation: range 40 -160 ng (majority 50 – 100 ng)
- 4 spiked tubes + 2 blank tubes per round
- 4 PT rounds per annum
- Z-score using trimmed median as assigned value (AV)
- Current SDPA = fixed at 7.5 % of AV

AIR PT sample production

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- Tubes thermally cleaned (< 1 ng benzene)
- Spiked in batches of up to 60 tubes from gas phase based upon procedures codified in ISO 6145 part 4
- 10 % of each spiked lot analysed at HSL (ISO 16017/17025)
- Homogeneity and stability results / requirements
  - < 1.5 % (via analytical measurements)
  - ~ 0.25 % (via mass flow considerations)
  - BTEX on tenax stability > 2 years (HSL/IRMM CRM studies etc.)
  - Thus meets ISO 13528 sample homogeneity and stability specifications for PT samples

AIR PT sample production

Performance over 10 recent PT rounds

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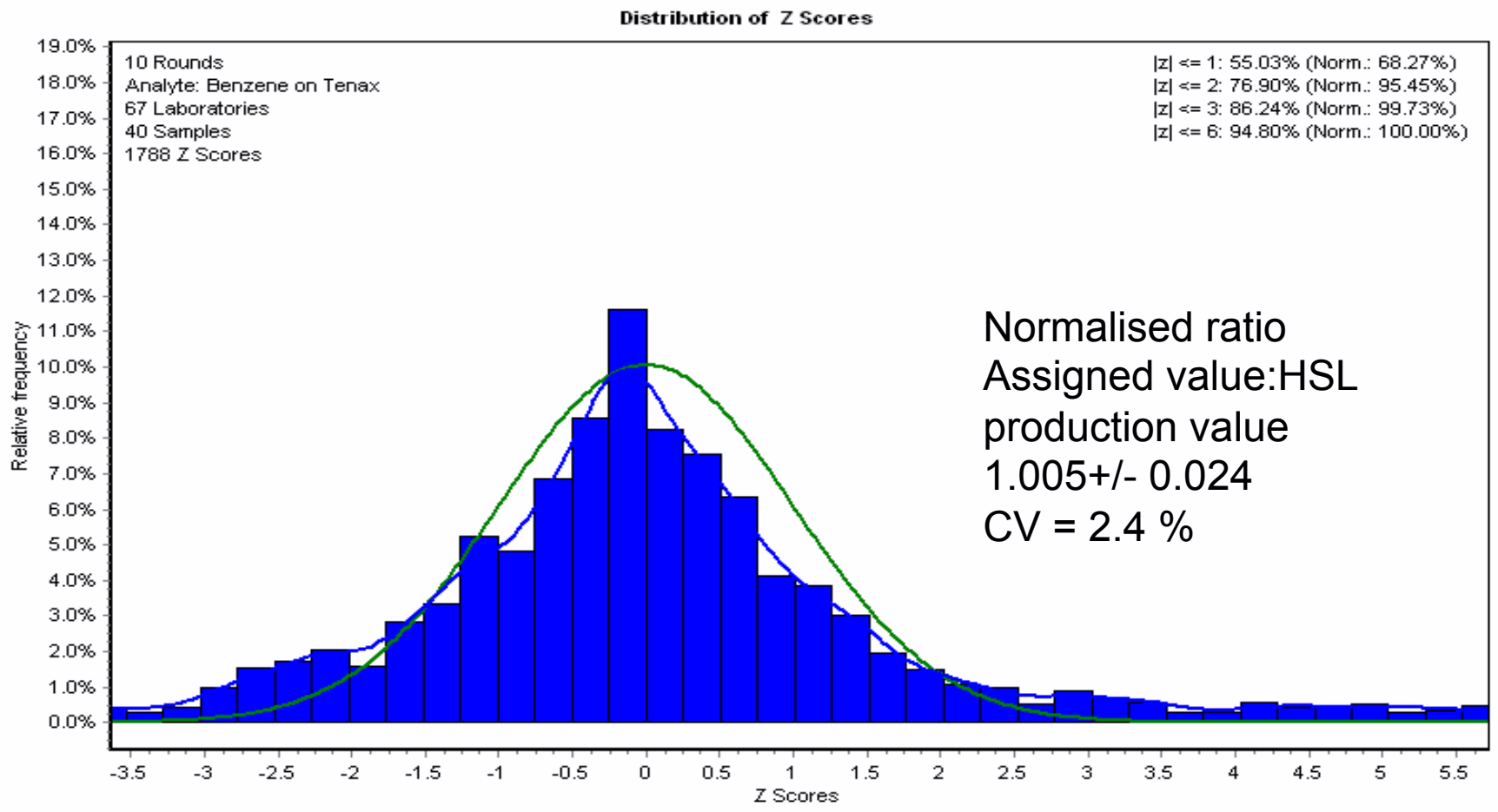

Performance over 10 recent PT rounds

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- Summary information
  - 67 participants in total
  - 55 participants took part in 4 or more rounds
  - 45 participants on average participated per round
  - 1788 z-scores calculated
  - 412 z-scores > 2 (23 % of submitted data)
  - 280 of these 412 z-scores (68 %) from laboratories where all four test samples per round were > 2

If this PT sample were to be a real sample?

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- Scenario
  - Diffusive sampling widely used as an indicative technique to support regulatory ambient air measurements (*in-situ* GC systems)
  - Lets assume that the PT sample becomes an ambient air diffusion sampler for 4 weeks
  - Equating to a loading of ~ 50 ng @ 1ppbv

Measurement Quality Objectives

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- EU MQO requirements
  - Expanded MU of +/- 30 % for indicative diffusion based measurements
  - Subtract MU component attributable to diffusive sampling (EN 13528)
  - Leaving an analytical MU contribution distilled as requiring consistent PT results within +/- 11.4 % of assigned value
- US MQO requirements
  - Laboratory bias < 20 % (acceptable)
  - Laboratory precision < 15 %

Summary of Results

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		EU	US
	n	Pass Rate	
Round 91	43	72 %	88 %
Round 92	46	57 %	78 %
Round 93	42	50 %	64 %
Round 94	40	63 %	65 %
Round 95	41	59 %	76 %
Round 96	44	64 %	80 %
Round 97	46	65 %	83 %
Round 98	47	68 %	81 %
Round 99	49	68 %	82 %
Round 100	49	69 %	76 %

Variability in laboratory performance ?

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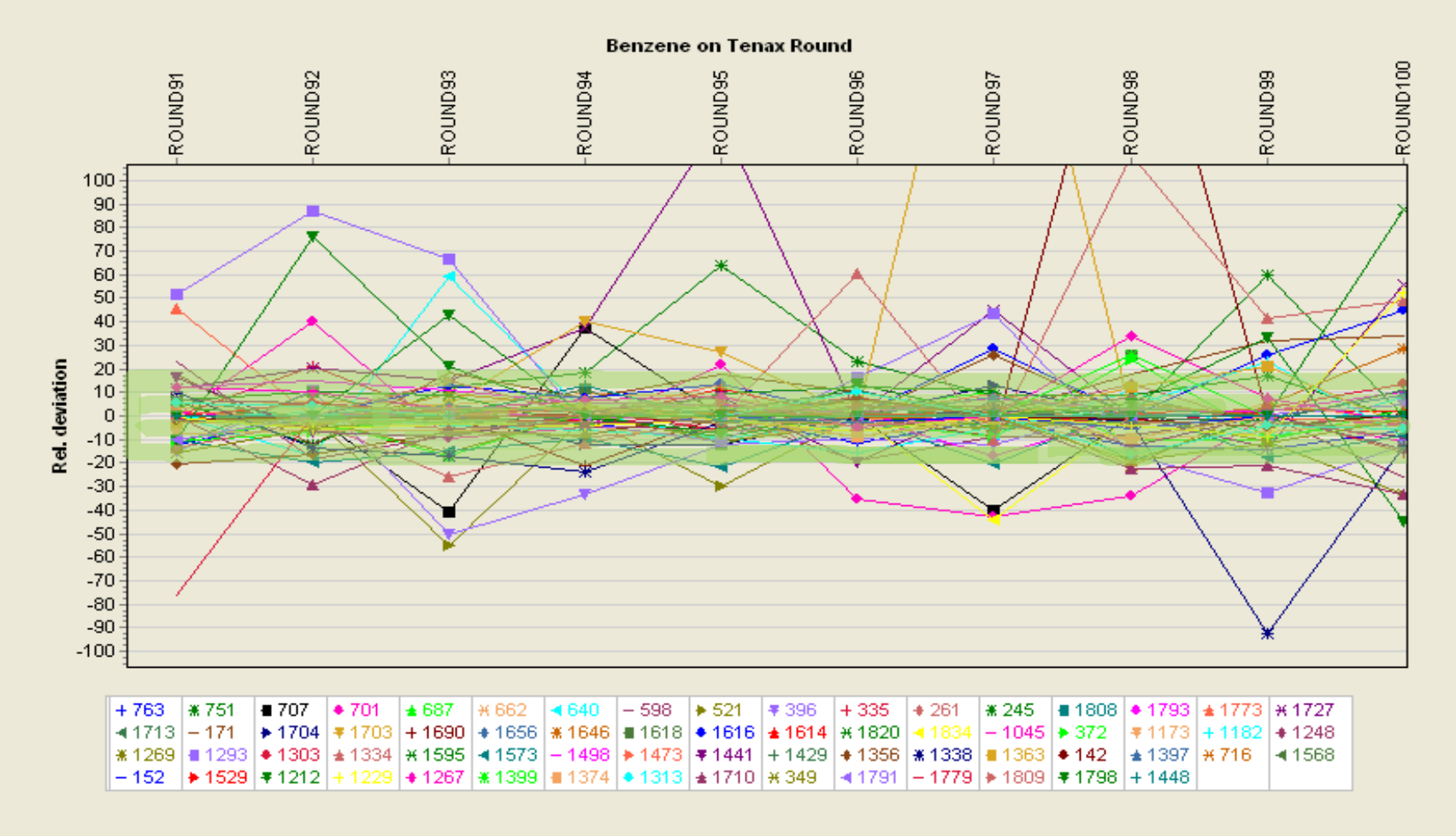

Laboratory A

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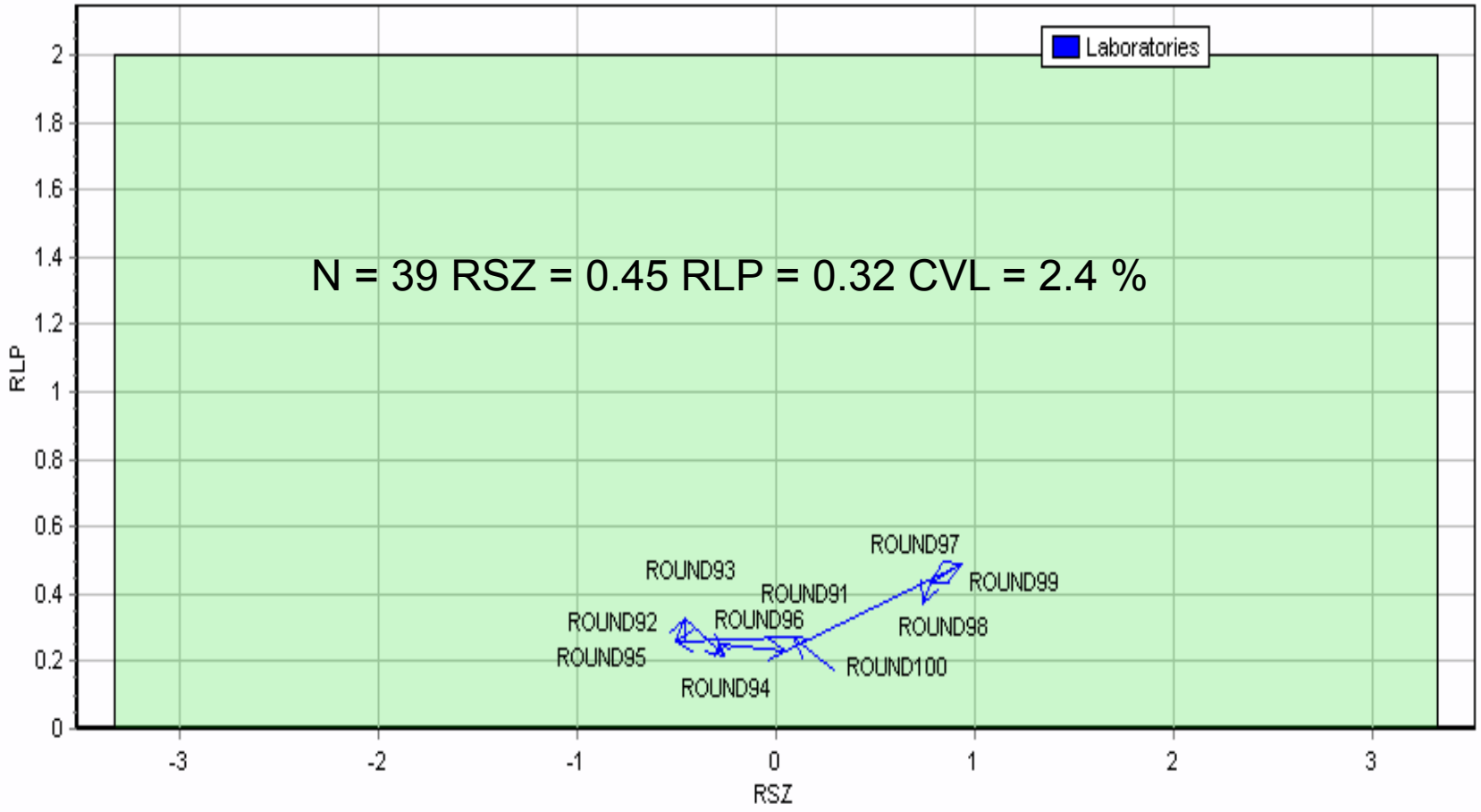

Laboratory B

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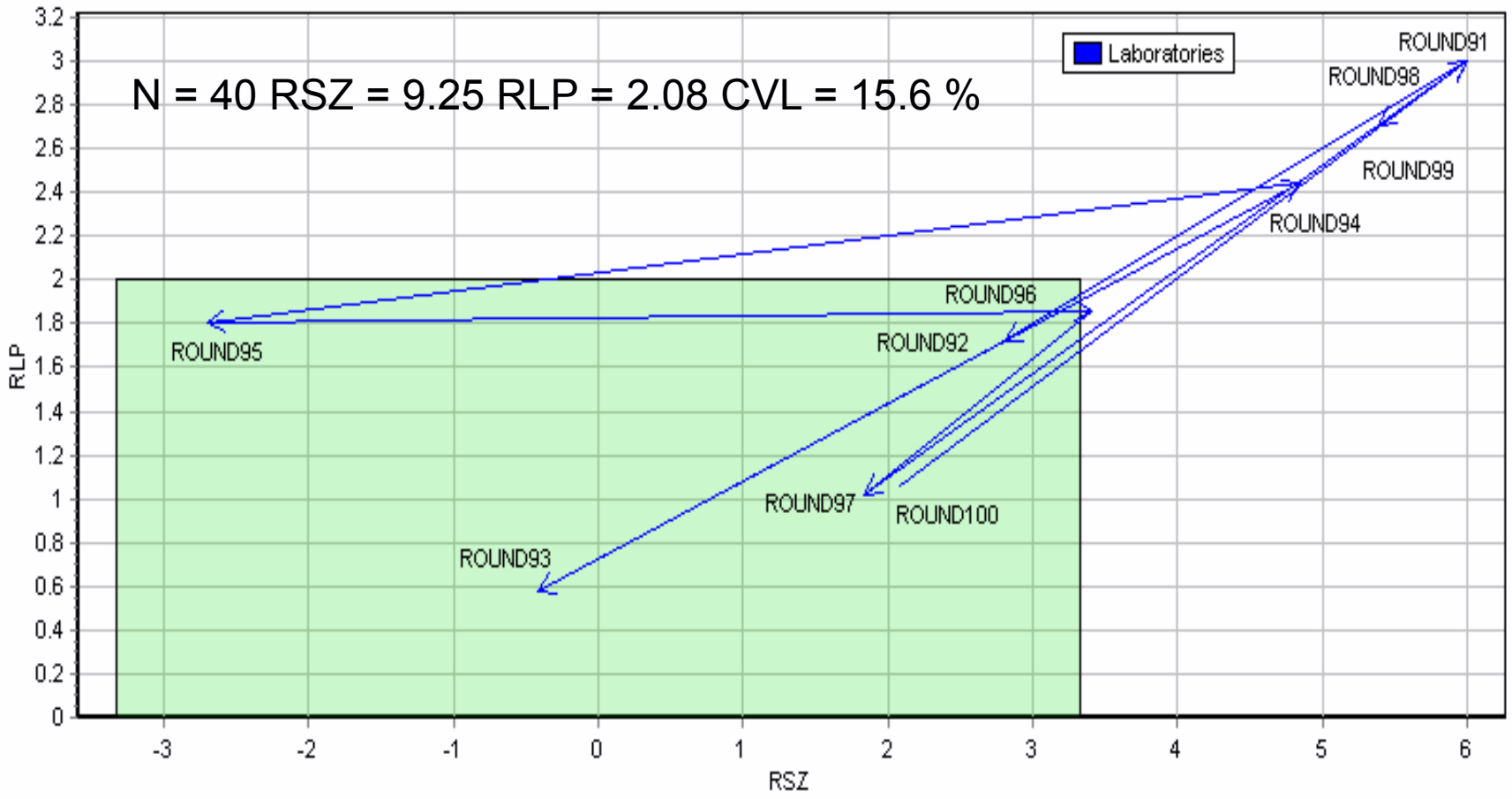

Conclusions (1)

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- Good TO-17 laboratory performance that meet respective MQOs is achievable
  - This required level of performance in expert laboratories undertaking trace analysis is demonstrable in PT participation/data
  - Pass rate for US MQO = ~ 80 % with a between lab round reproducibility of ~ 10 %
- For laboratory not (consistently) meeting MQOs
  - Need to undertake root cause analysis when poor PT results become apparent
  - Anecdotal evidence from PT feedback that instrument care and calibration are major factors in some laboratories

Conclusions (2)

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- PT data reporting tools (trending) useful to assist labs to monitor performance over time
- Need for QC and RMs to supplement PT activities
- Provision of more realistic and challenging PT samples

Research Interests

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- Better understanding of variation in PT data over time
  - Collating more contextual information – better feedback
  - Human factors in PT performance?
    - Staffing issues - turnover, leadership, rosters, on-call expertise?
    - Perception of quality - measurement climate in the laboratory?

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