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Performance of laboratories undertaking VOC in Air Analysis using Thermal Desorption

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Overview of Presentation

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- Background
- Performance of laboratories undertaking VOC analysis using TD technique as seen through proficiency testing (PT) studies
- PT Performance requirements
- Future requirements /challenges for PT

HSL overview

HSL is part of the science division of the UK Health and Safety Executive, HSE and supports their regulatory mission in protecting workers by supplying scientific, engineering and forensic capabilities and services. HSL also delivers high quality science serving the needs of wider industry and government both in the UK and overseas.

This includes developing measurement science (published validated methods) for the determination of pollutants in air and providing supporting quality assurance tools (proficiency testing, reference materials).

What is proficiency testing?

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Proficiency testing comprises an interlaboratory system for the regular testing of the accuracy that the participant laboratories can achieve. In its usual form, PT scheme organisers distribute homogeneous and stable test materials to each of the participants, who analyse the materials under typical conditions and report the results to the organisers. The organisers compile the results and inform the participants of the outcome, usually in the form of a score relating to the accuracy of the result.

AIR proficiency testing overview

- The AIR proficiency testing (PT) scheme, a merger in 2014 between the long standing HSL WASP PT and LGC (UK Designated Institute for Chemical Metrology) STACK PT schemes.
- Offers a wide range of chemical PT samples covering ambient, fence line, indoor, workplace and stack air (grab) monitoring applications for those activities that involve returning samples to a laboratory for testing.
- Scheme is accredited to ISO 17043.

AIR PT Sample 12:Overview

TD tubes spiked with BTEX (Tenax/Carbopack X)

Spiked in the range 50 – 1000 ng

Typically spiked < 250 ng

4 spiked tubes + 2 blank tubes per round

4 PT rounds per annum

Sample 12 started in 1997

Aimed at laboratories undertaking sampling to procedures set out in EPA TO-17, EPA 325, ISO 16017, ISO 16000-6 etc.

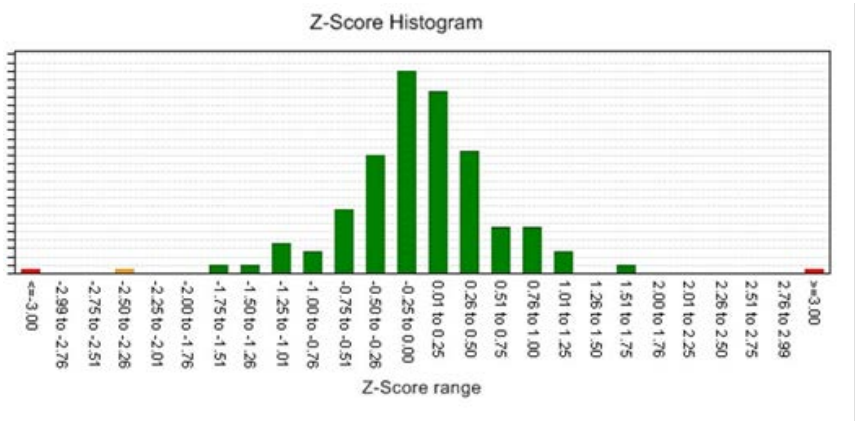
AIR PT Scoring

$$z\ score = \frac{(x - X)}{SDPA}$$

x = the result reported by the participant

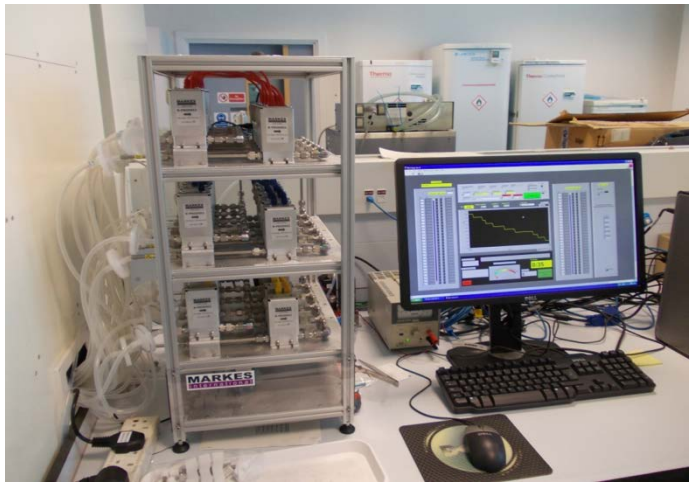
X = the assigned value (trimmed median)

SDPA = standard deviation for proficiency assessment



$\|z\|<2$	Satisfactory (95%)
$2<\|z\|<3$	Questionable (5%)
$\|z\|>3$	Unsatisfactory (0.3%)

AIR PT Sample 12:Production



- Tubes thermally cleaned (< 1 ng benzene)
- Spiked in (repeat) batches of up to 60 tubes from gas phase based upon procedures codified in ISO 6145 part 4
- 10 % of each spiked lot randomly analysed at HSL
- ISO 16017 method that is ISO 17025 accredited

AIR PT Sample Homogeneity + Stability

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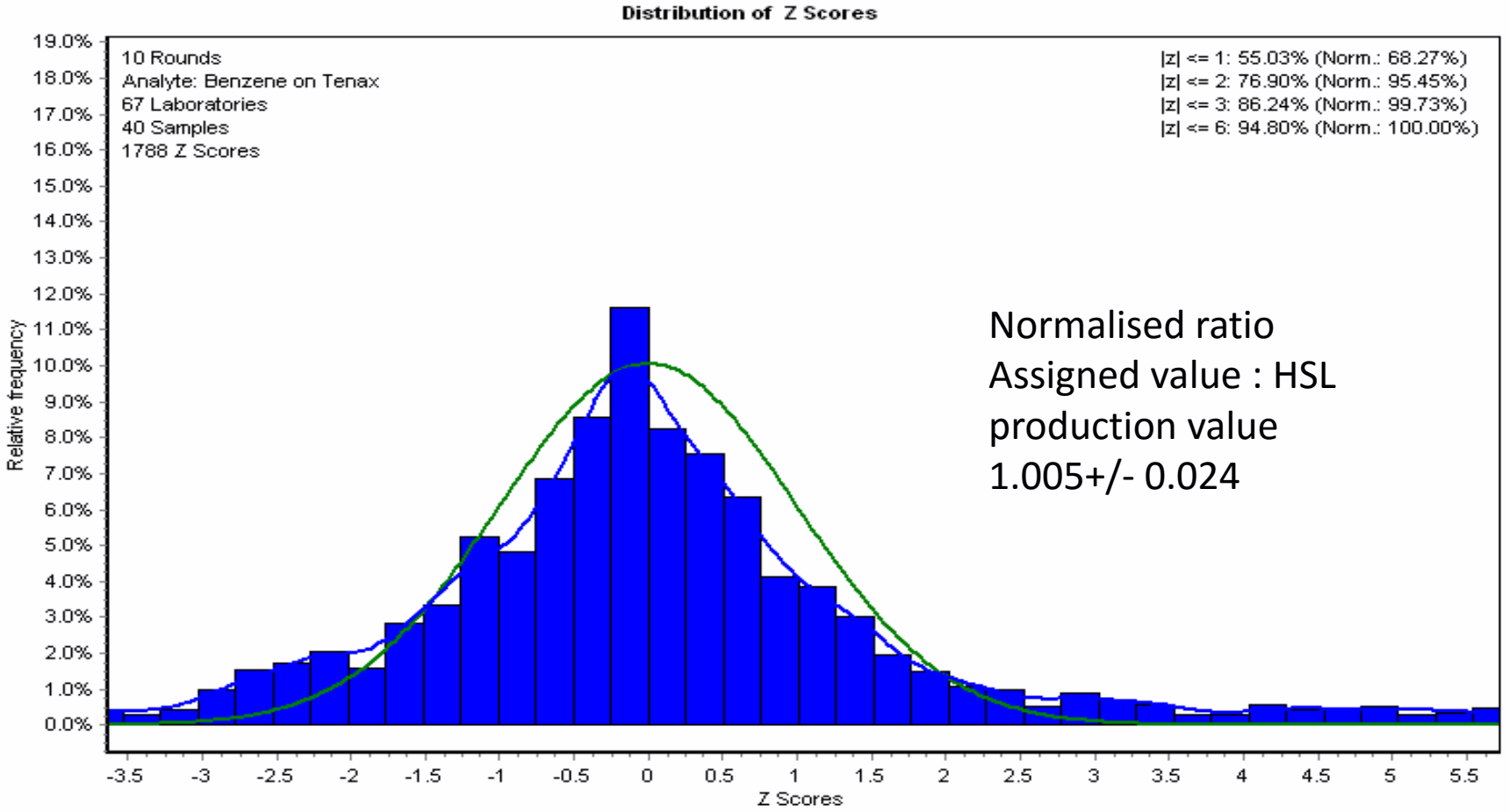
- Homogeneity
  - ≤ 0.3 x SDPA (ISO 13528)
  - Sample 12 SDPA = 7.5 %
  - Therefore homogeneity must be ≤ 2.5 %
  - < 1.5 % (via GC-TD analytical measurements)
  - < 0.5 % (via mass flow measurements)

- Stability
  - Stability $t_1 - t_0 \leq 0.3$ x SDPA (ISO 13528)
  - Sample 12 SDPA = 7.5 %
  - Therefore stability must be ≤ 2.5 %
  - BTEX on sorbent stability > 2 years (HSL/IRMM CRM studies)

AIR PT Sample 12: Group results



AIR PT Sample 12: Group results

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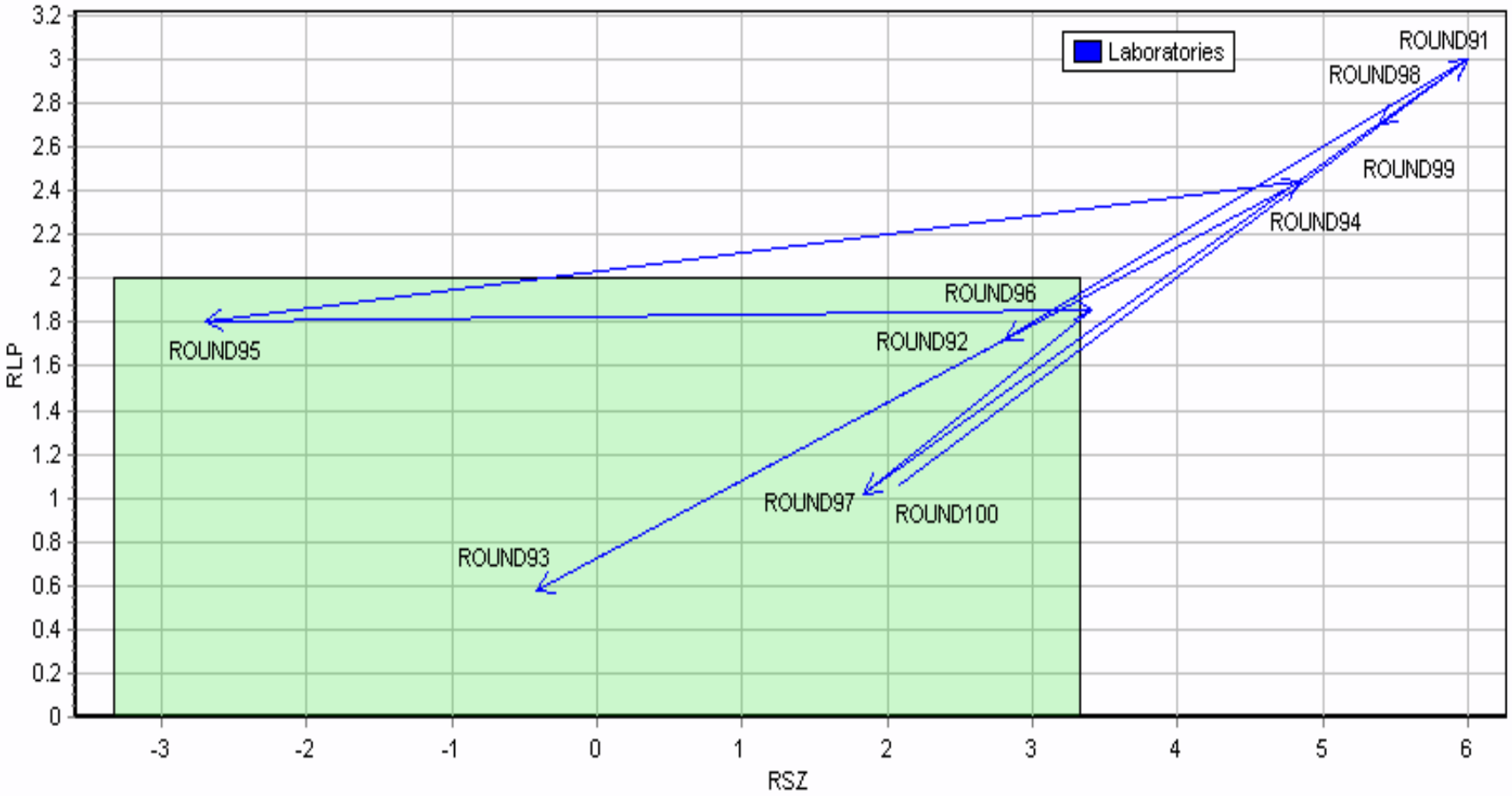
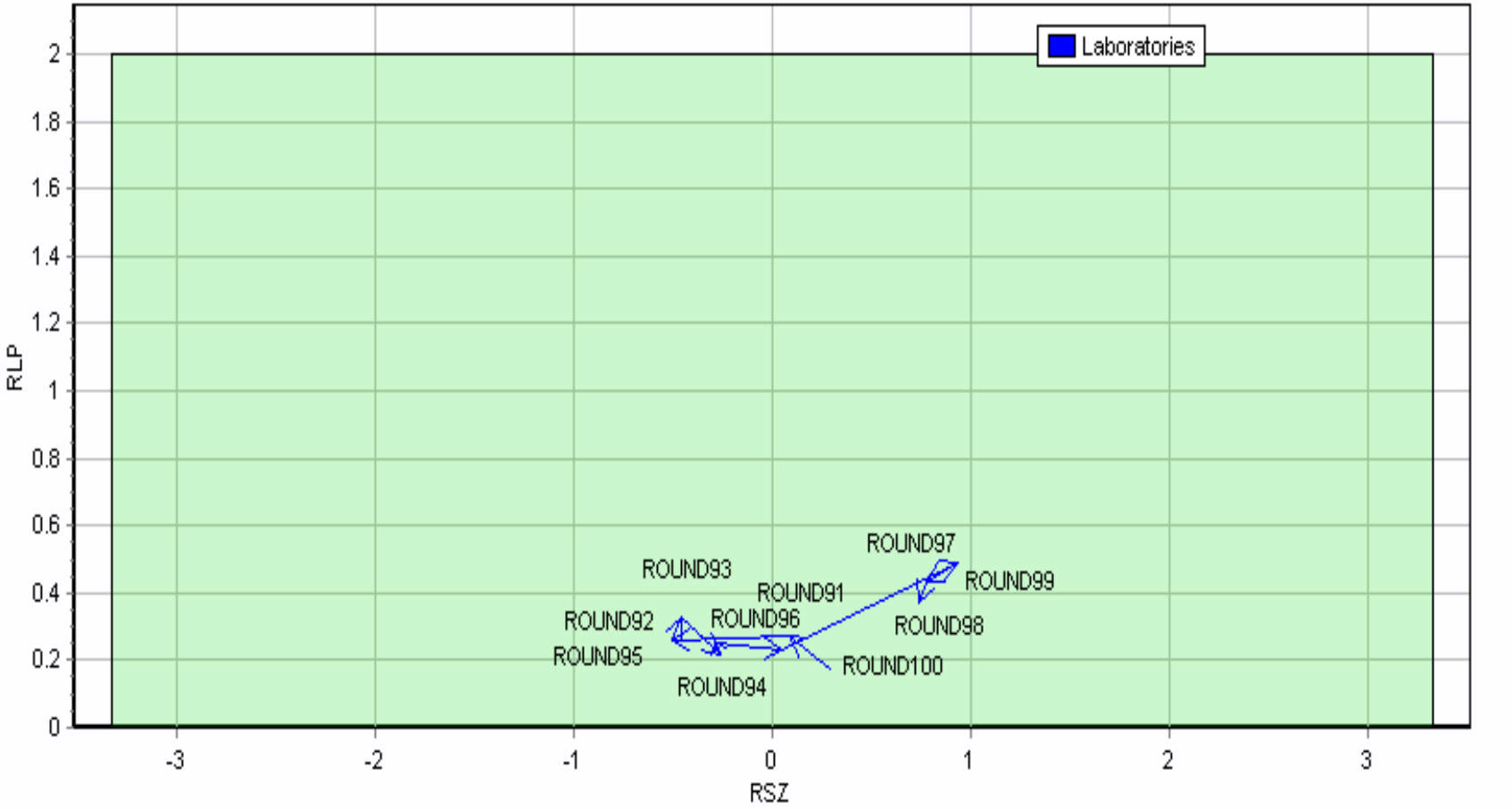
- 1788 z-scores calculated
- 412 z-scores > 2 (23 % of submitted data)
- 280 of these 412 z-scores (68 %) from laboratories where all four test samples per round were > 2
- Calibration, trap and contamination issues

AIR PT Sample 12: Individual results



AIR PT Sample 12: Individual results



AIR PT Sample 12: Results in Context

- 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 is a “ real” ambient air sample
  - 2 week diffusive air sampler that collected say ~ 100 ng

AIR PT Sample 12: Results in Context

- 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 %

AIR PT Sample 12: Results in Context

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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 %

Ongoing / future challenges for PT

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- PT scoring targets need to reflect "fitness for purpose"
  - We are considering tightening the SDPA value for Sample 12
  - Challenge: Homogeneity/stability assessment *v* analytical imprecision
- More realistic matrix-matched PT samples
  - Real-air PT sample for Sample 12?
  - More challenging for particle on filter PT samples!!!
- Handling measurement uncertainty (ISO 17025)
- Sampling PT?

Acknowledgements and Contact Info.

Thanks to the conference organisers for this opportunity to present

Thank you for your attention

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https://www.lgcpt.com