



Excellence through measurement



Environmental Day 2011: LGC Standards, Proficiency Testing

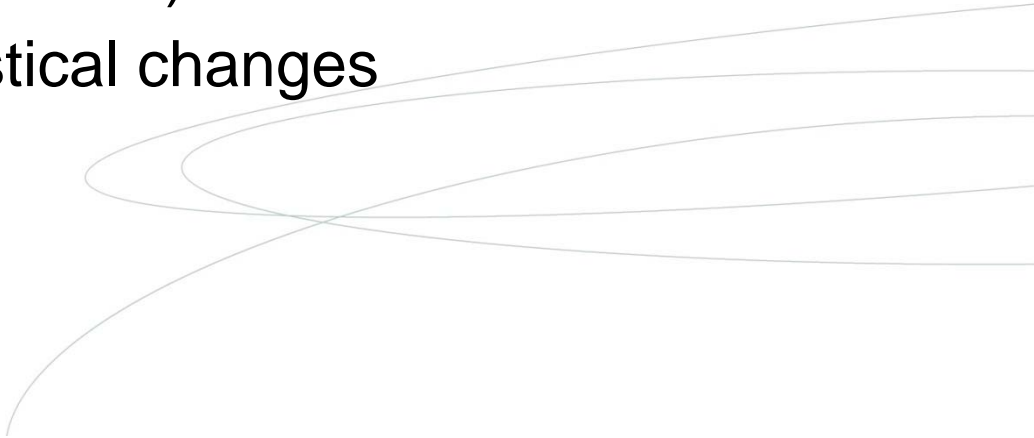
CONTEST PT scheme review

Matthew Whetton

CONTEST: Introduction

- CONTEST
 - Methods survey 2010
 - Scheme performance
 - Results of trial rounds in 2011
 - Future developments
- 

CONTEST: 2011 - 2012

- Rounds
 - 2011/2012 Five rounds (Rd 76 - 80)
 - Samples
 - Sample E for WAC Leaching (EN 12457)
 - Trial sample for BTEX
 - Sample 4 (PAH in MeOH) was withdrawn
 - No significant statistical changes
- 

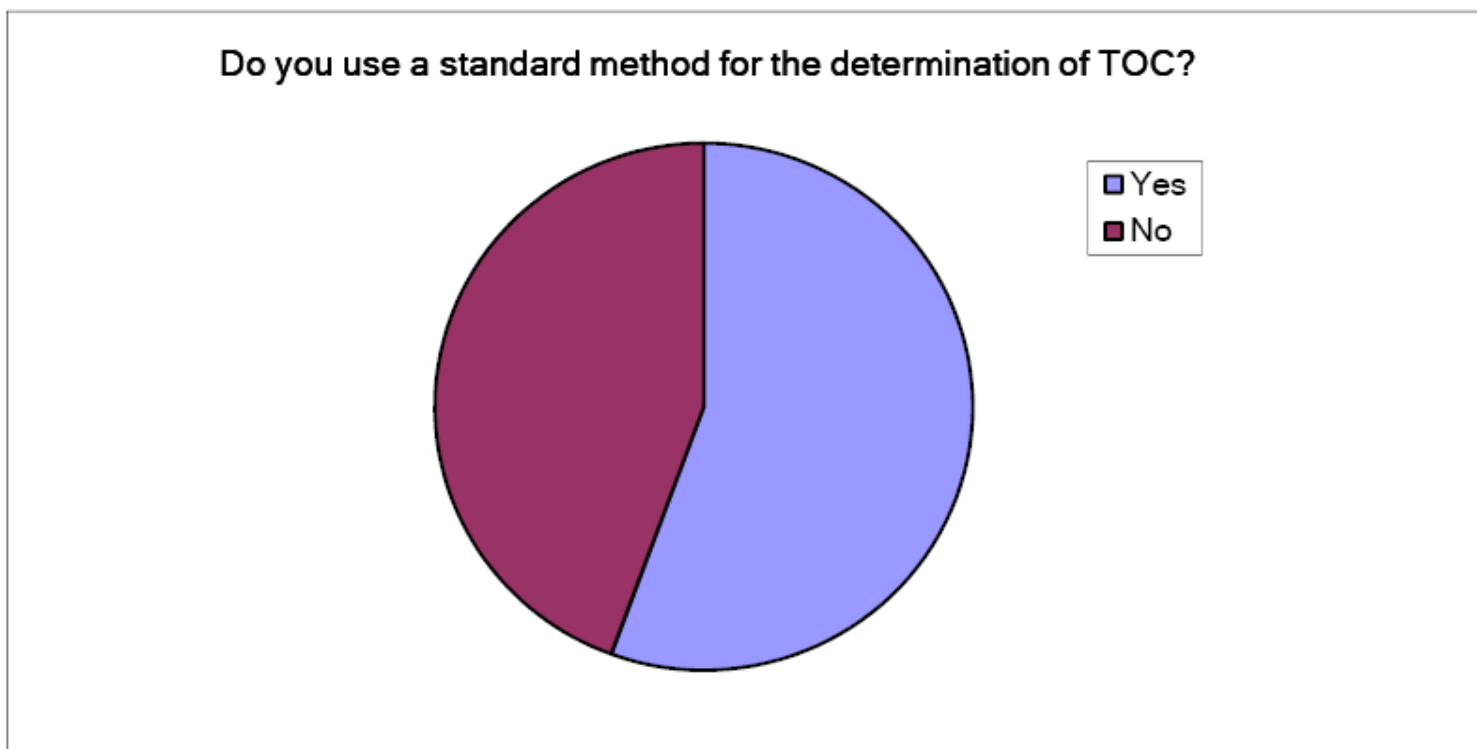
CONTEST development (2010)

- A questionnaire was sent out:
 - Leaching samples:
 - Listing possible scheme structures
 - Identifying leaching methods
 - Identifying key the analytes
- The method information was sought regarding:
- Sample 3c TOC
- Sample 3c TPH
 - Where appropriate the method information will be applied for 2012/13

CONTEST: methods survey (TOC)



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- **Methods listed**

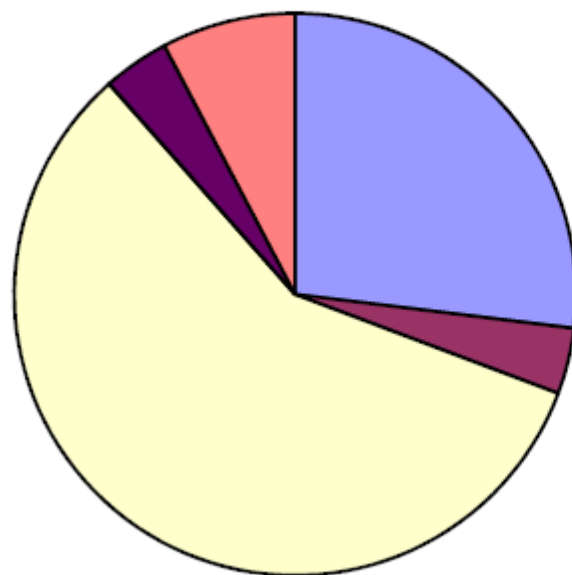
- BS 1377, EN 13137 (2001), USEPA 9060, CNR IRSA 5 Q 64 Vol 3 1988, BM019, NF ISO 10694, BS7755-3.8, D.M. 1309/1999

CONTEST: methods survey (TOC)



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Which of the method classes below most accurately describes the method you routinely use?

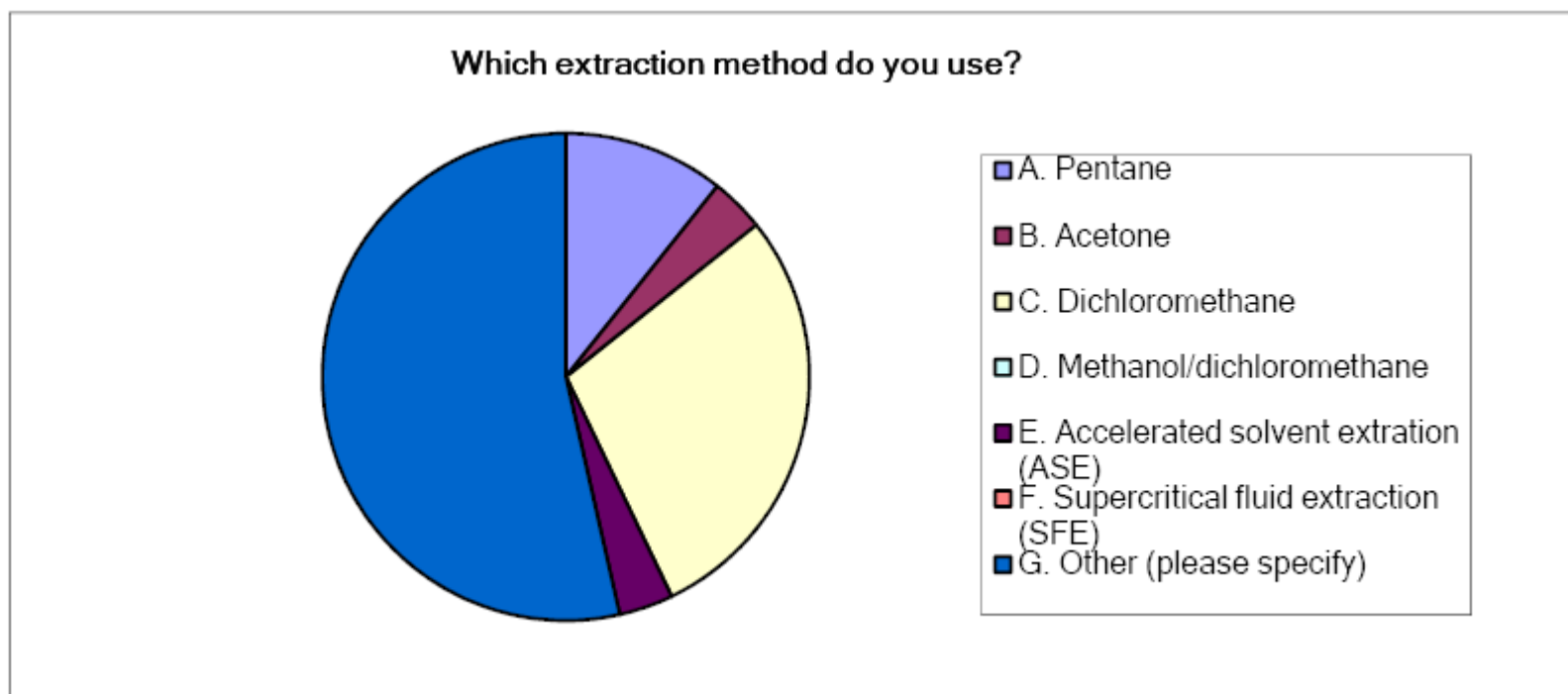


- A. Manual, Walkley-Black method
- B. Manual, Other chemical oxidation method
- C. Instrumental, Combustion method
- D. Instrumental, Chemical oxidation
- E. Non-destructive method
- F. Other (please specify)

CONTEST: methods survey (TPH)



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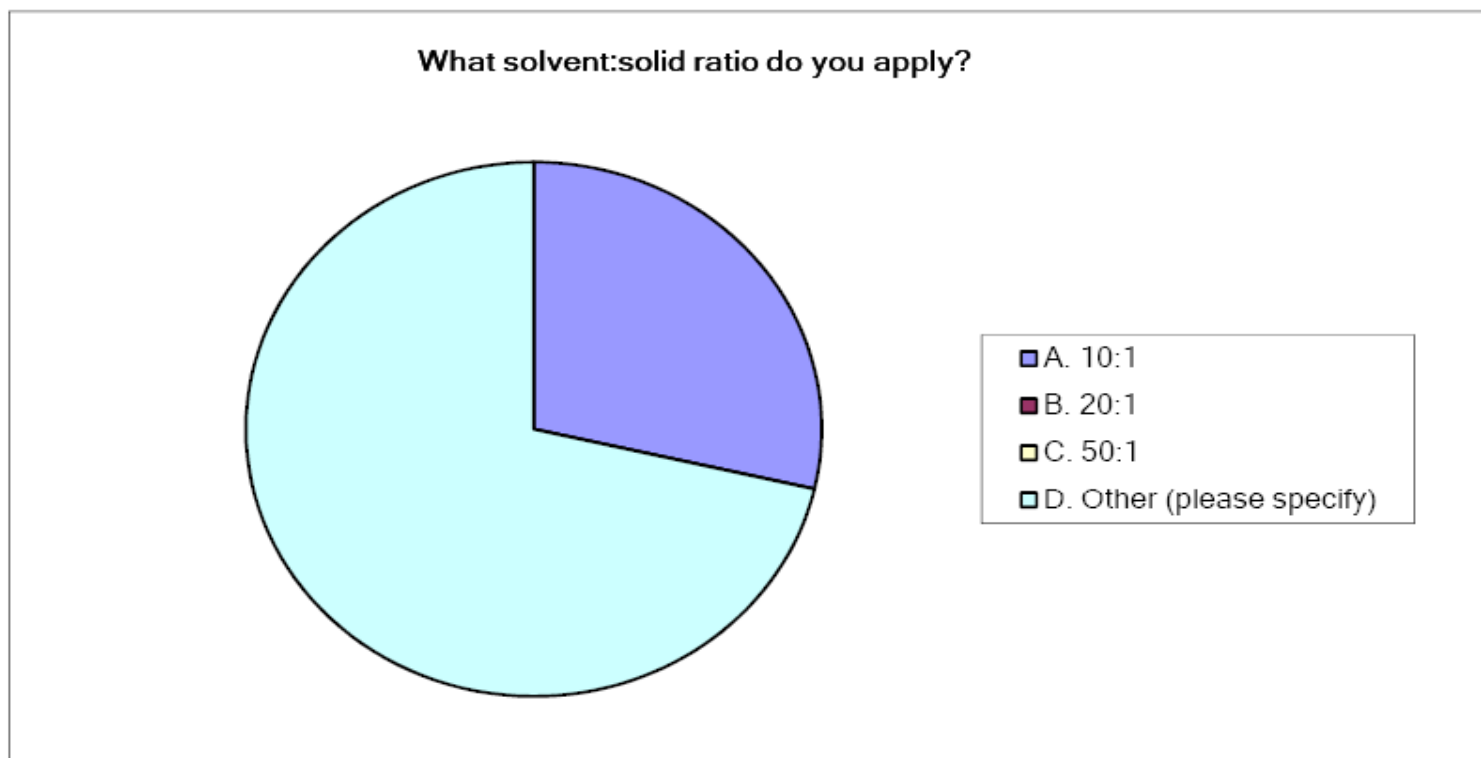


- 'Other' methods listed
 - Pentane/Acetone, Hexane/Acetone (1:1, 2:1), Heptane/Acetone, DCM/Hexane

CONTEST: methods survey (TPH)

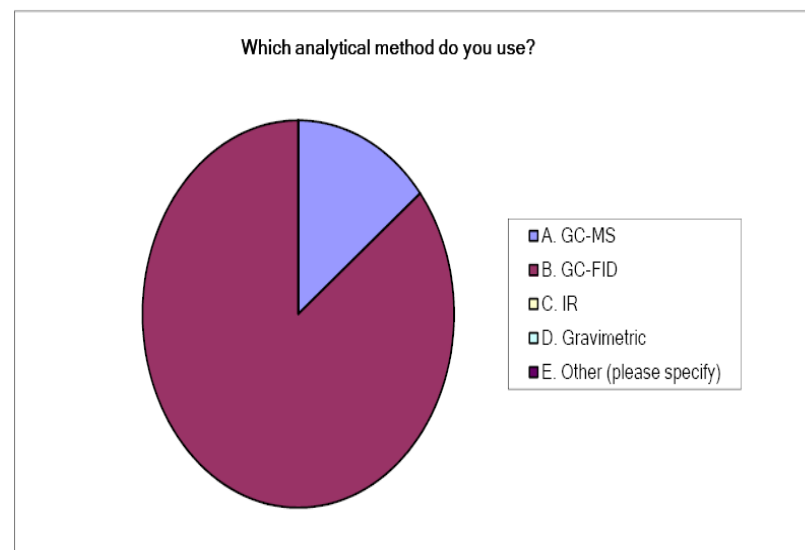
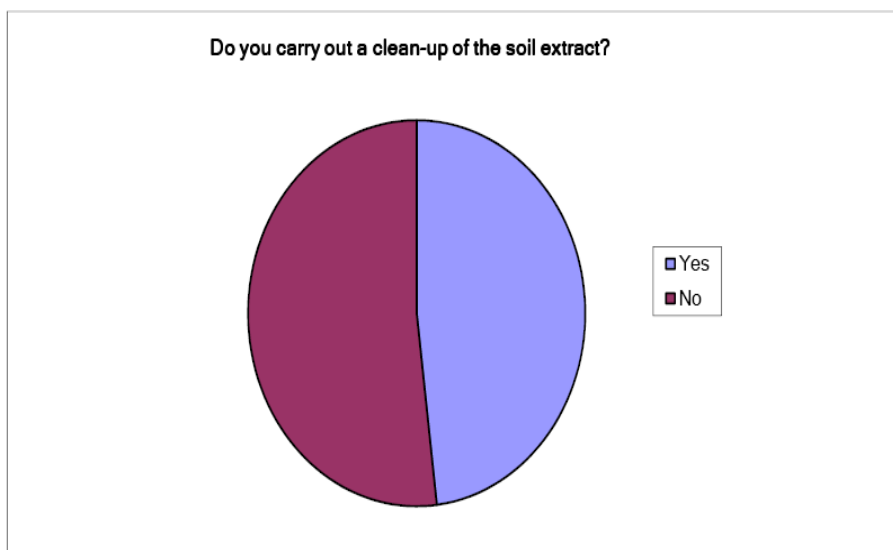


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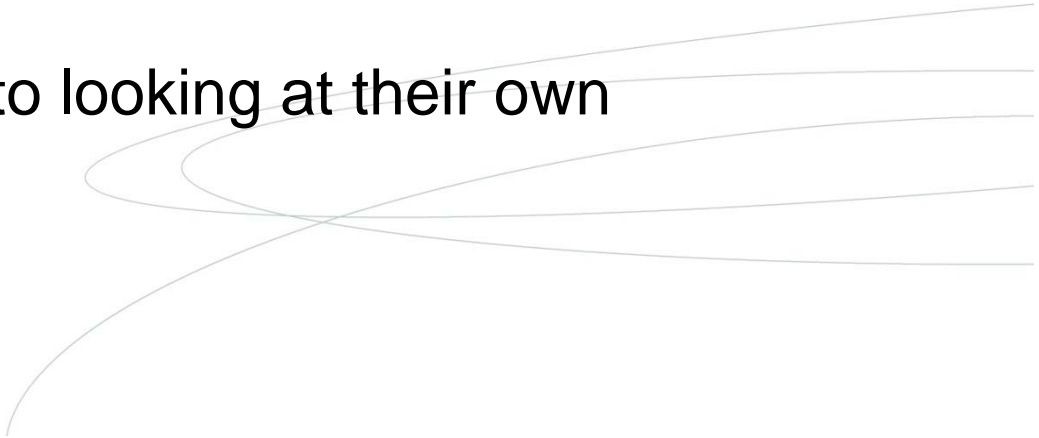
- 'Other' solvent:solid ratios listed
 - 0.5:1, 1:1, 2:1, 2.5:1, 3:1, 5:1, 8:1, 30:1

CONTEST: methods survey



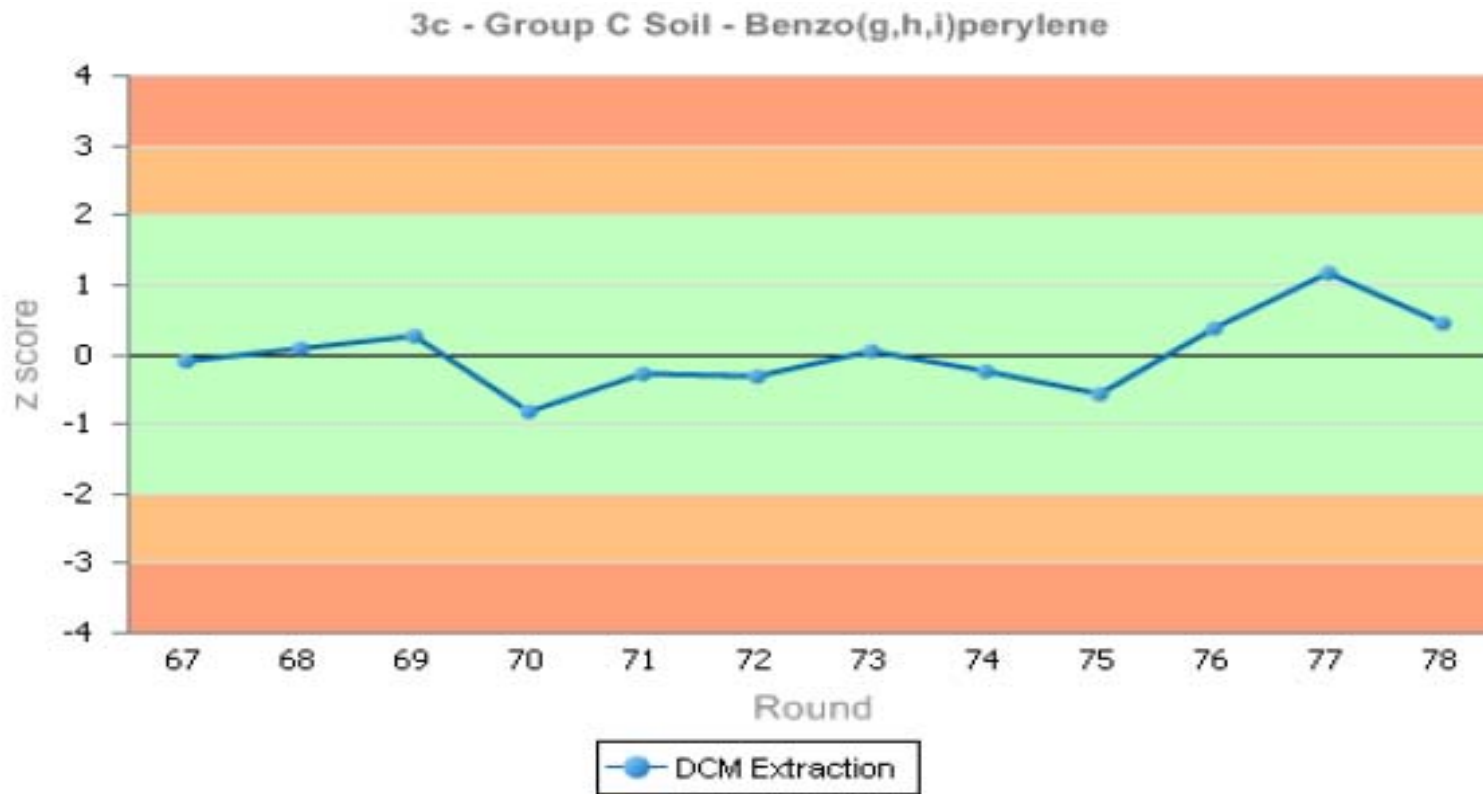
- ‘Clean-up’ methods listed
 - SPE:Florisor, Sodium sulfate:silica 60, Silica gel, sodium sulfate:florisil, florisil

CONTEST: Scheme performance

- PORTAL now contains a large amount of data
 - Many studies have shown performance improves over time
 - How has CONTEST progressed since PORTAL was introduced?
 - Participants are used to looking at their own performance
- 

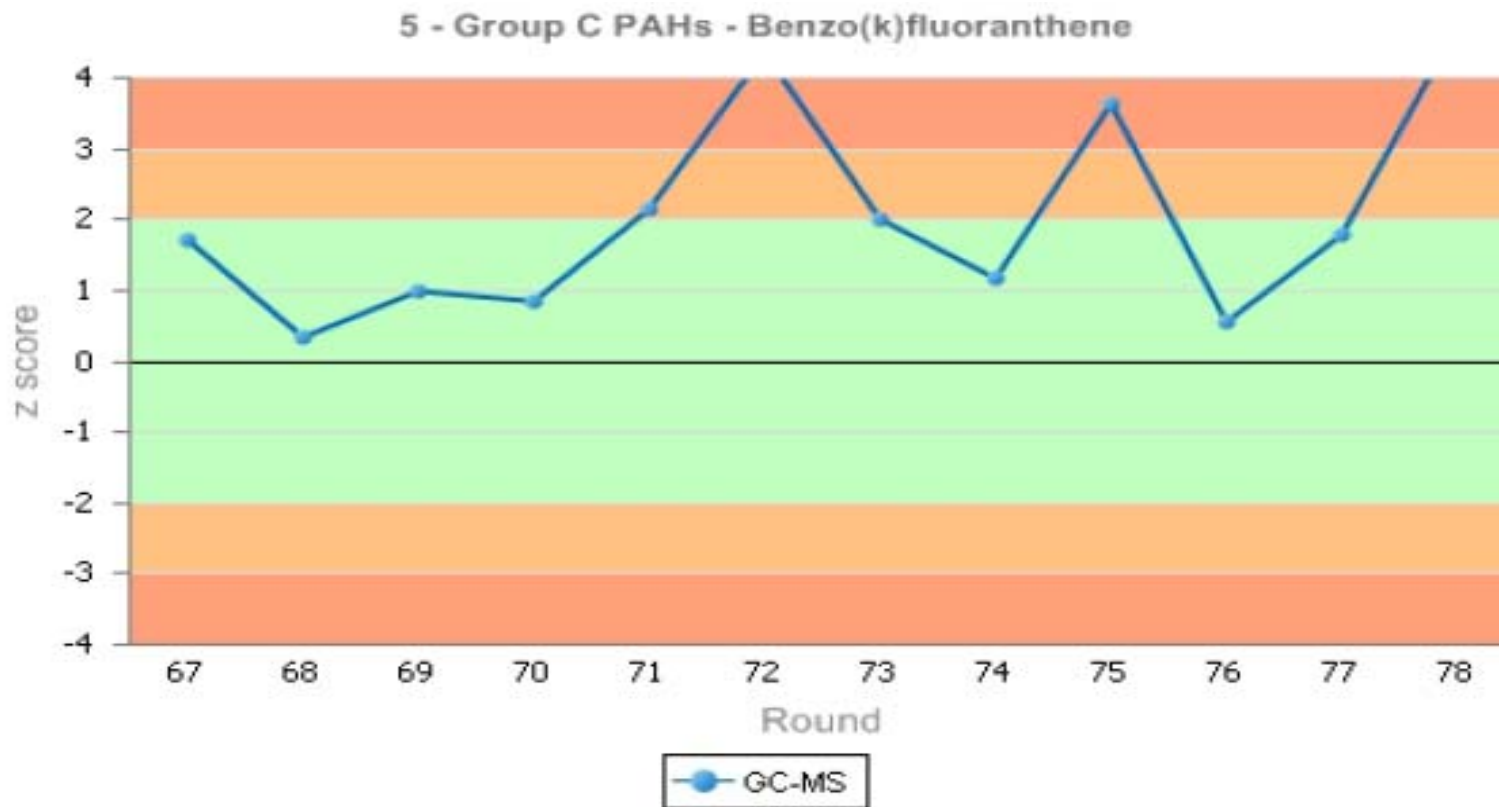
CONTEST: Individual performance

- When performance is good



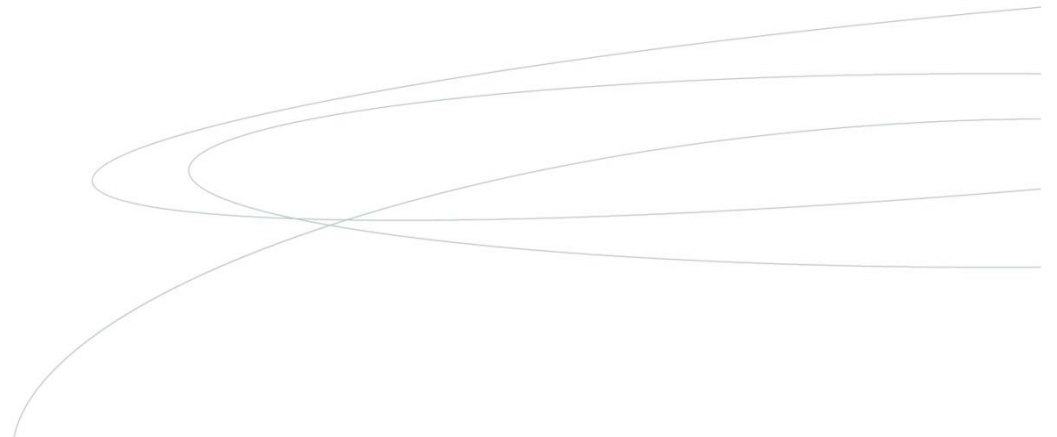
CONTEST: Individual performance

- When performance is 'not so good'



CONTEST: Scheme performance

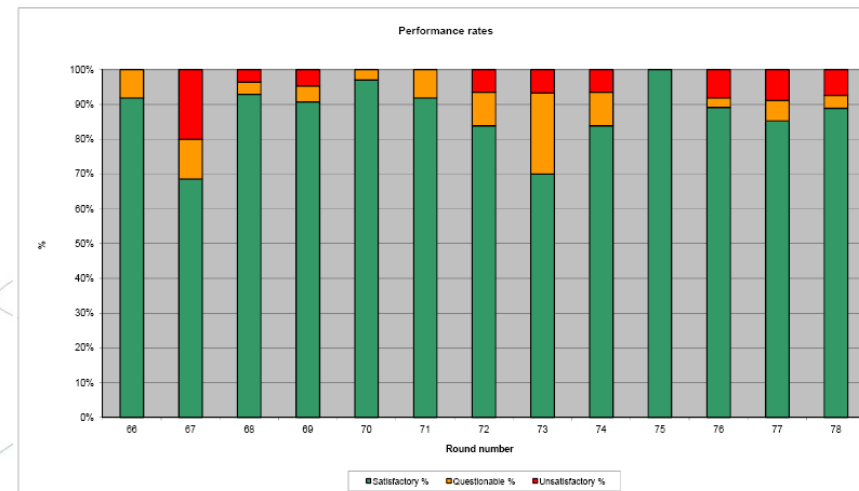
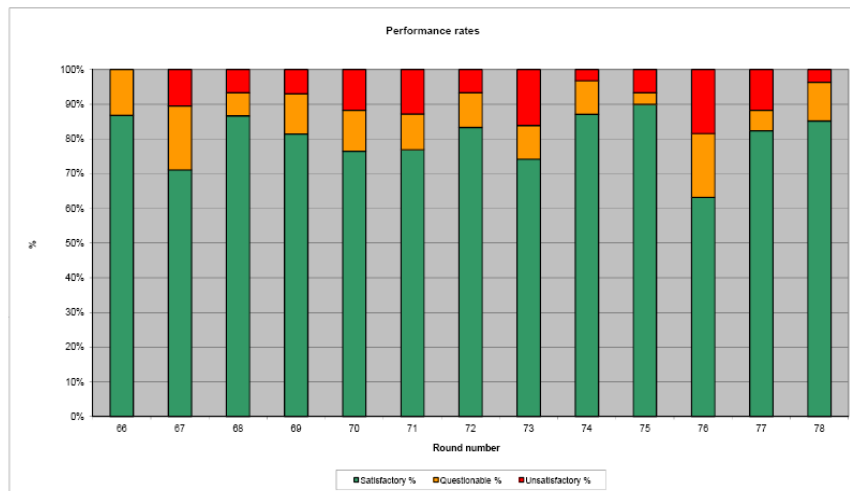
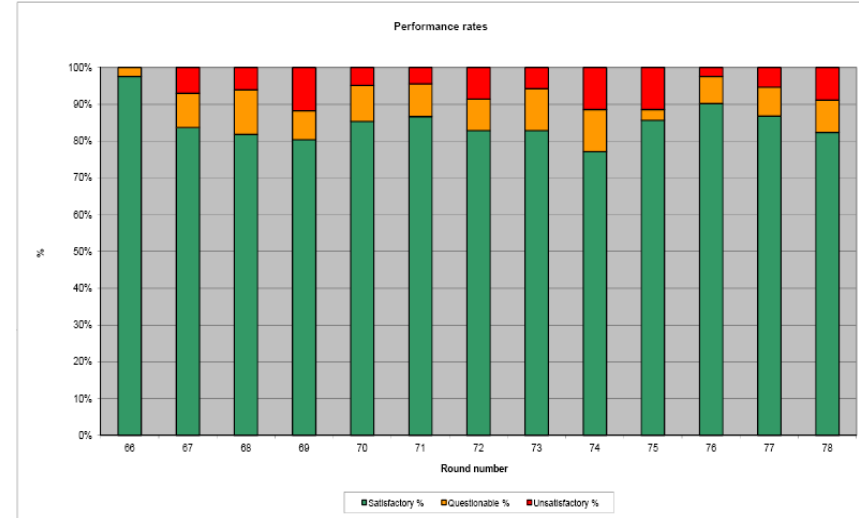
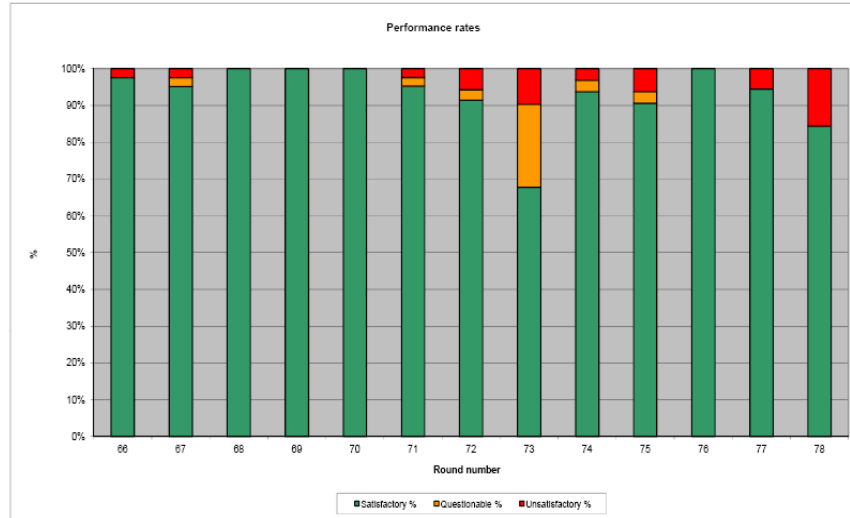
- Performance measures
- % Satisfactory performance
 - Consistent statistical assessment (AV determination, SDPA)
 - Relatively consistent participant group
- Spread of data
 - Robust assessment, relatively unaffected by outliers



CONTEST: Sample A (As, Cr, V, Co)



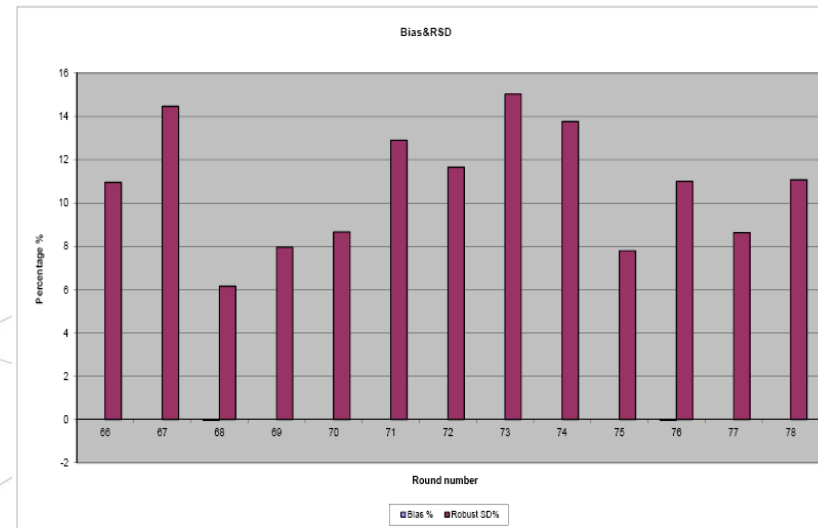
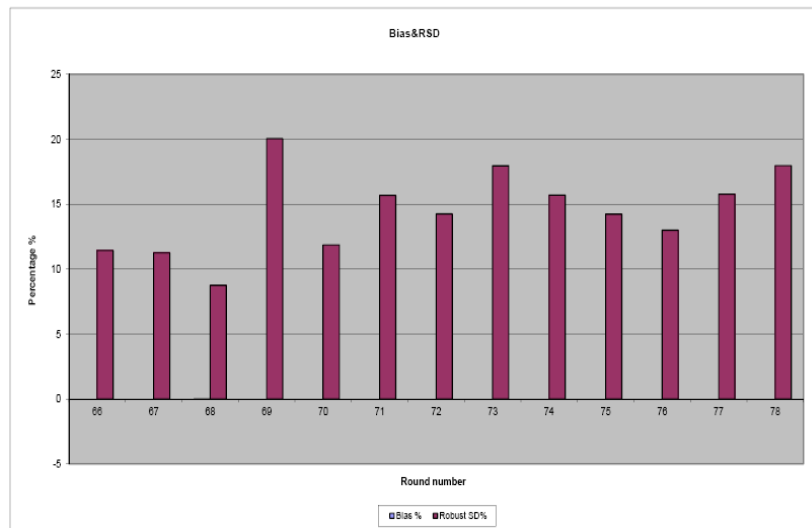
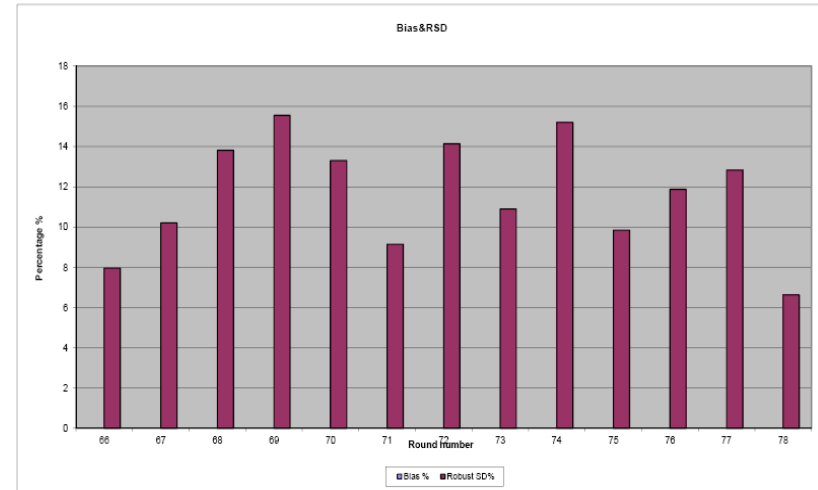
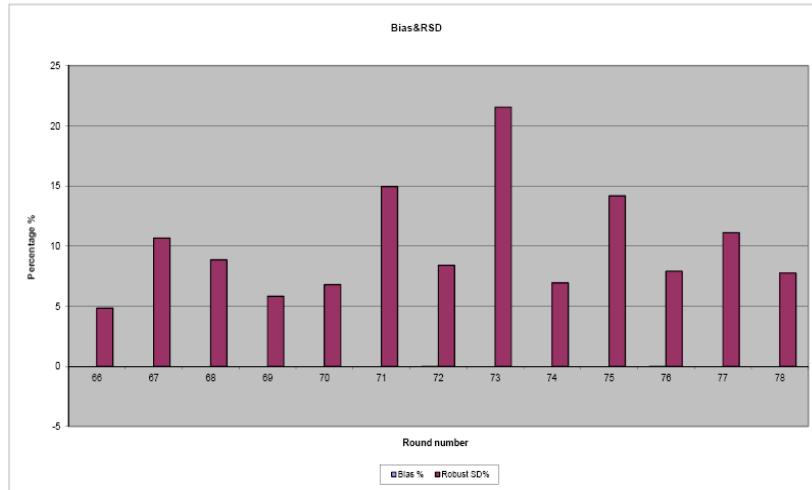
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CONTEST: Sample A (As, Cr, V, Co)



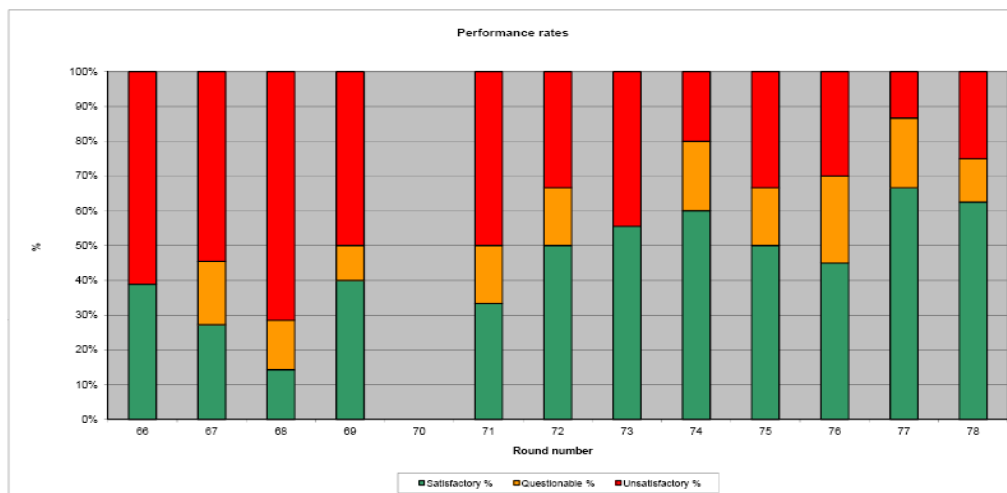
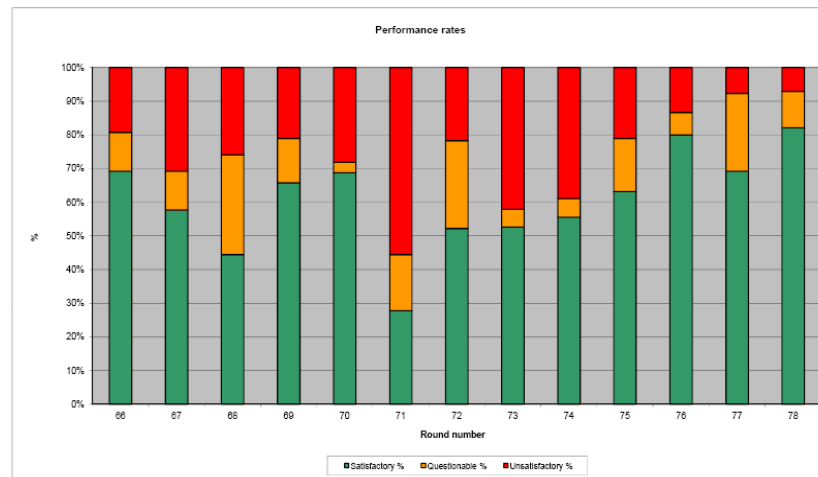
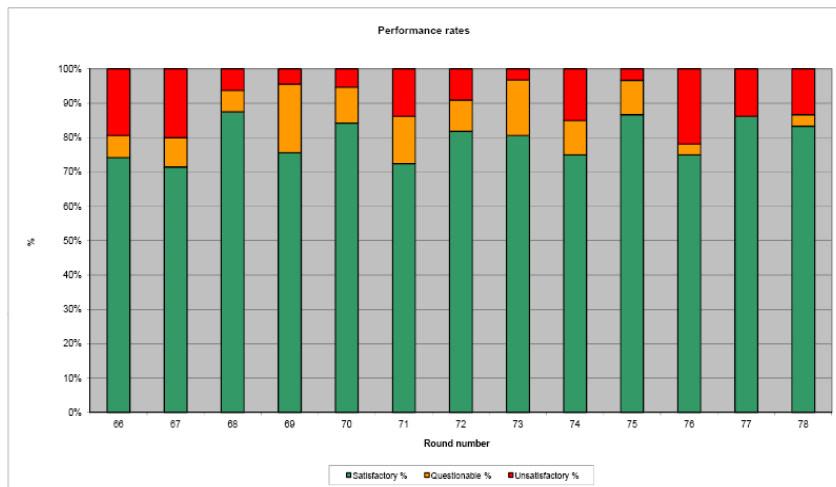
Excellence through measurement



CONTEST: Sample A (Hg, Se, Cr VI)



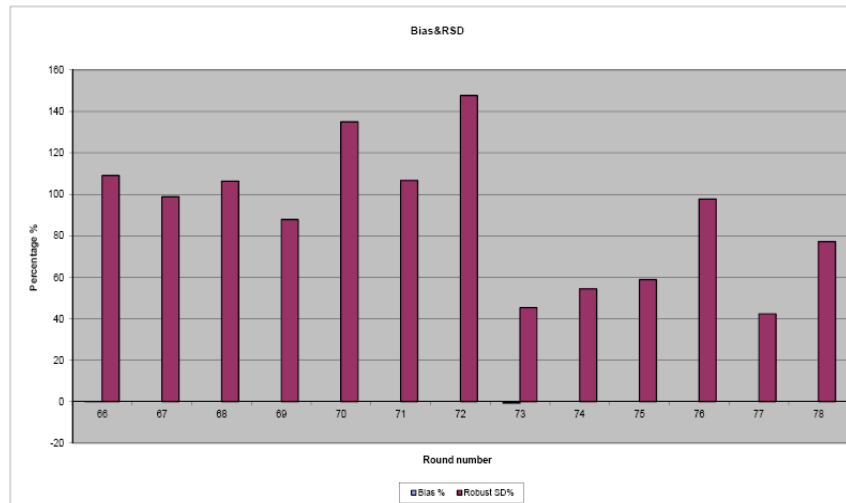
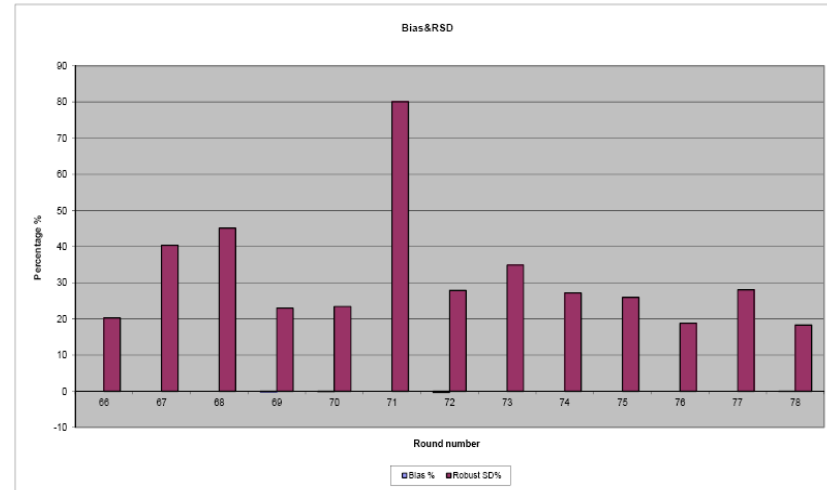
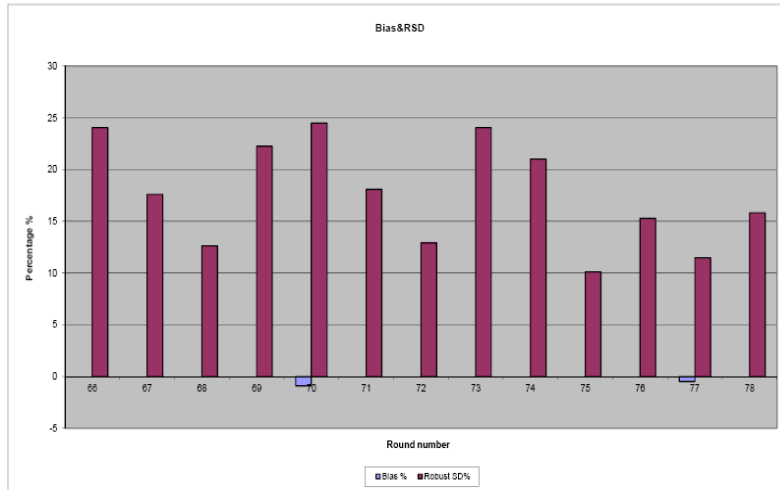
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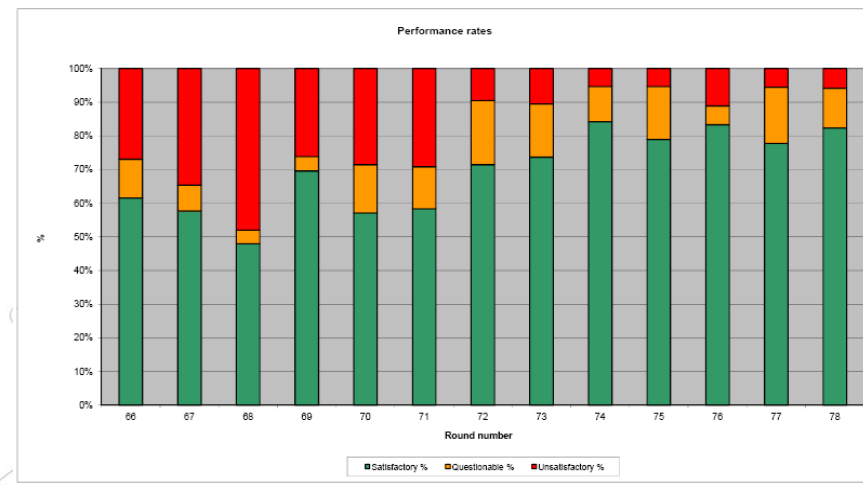
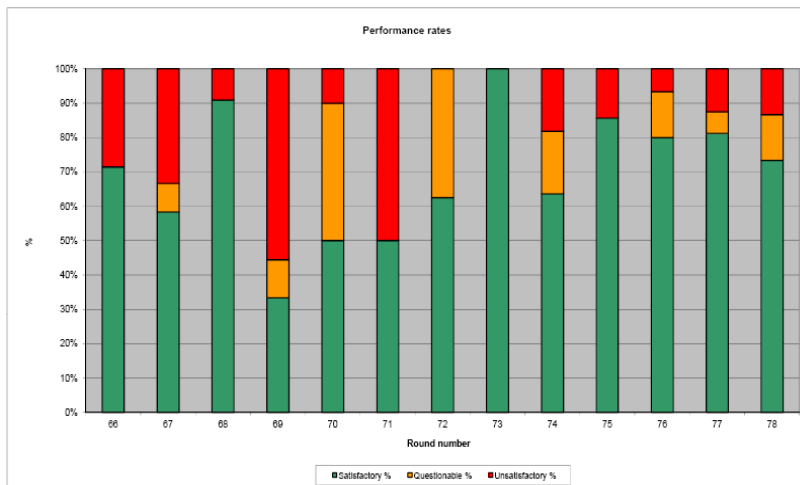
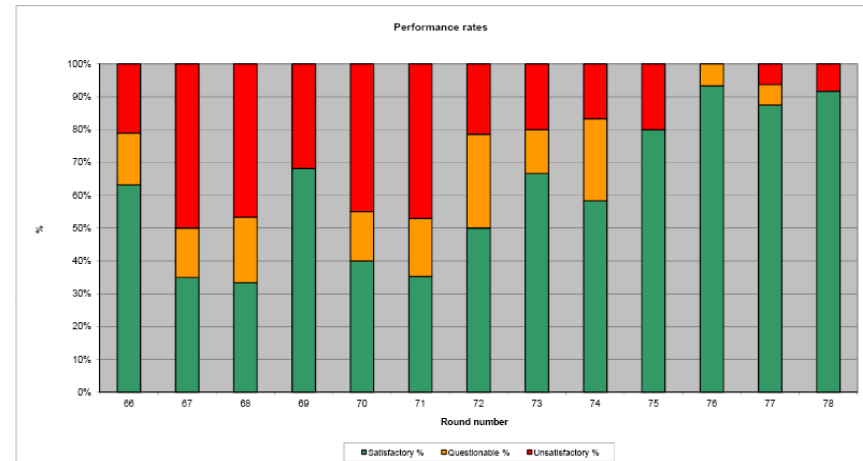
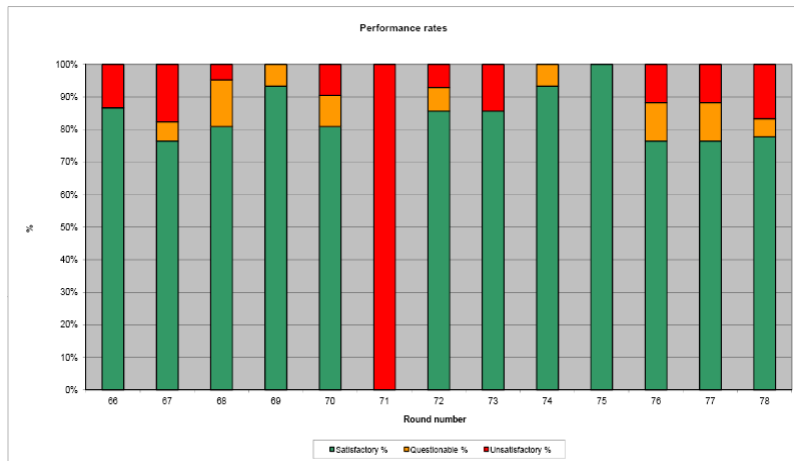
CONTEST: Sample A (Hg, Se, Cr VI)



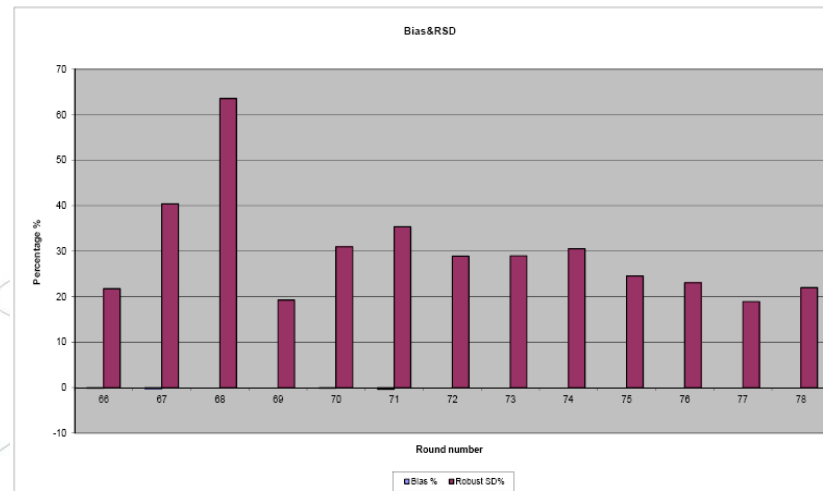
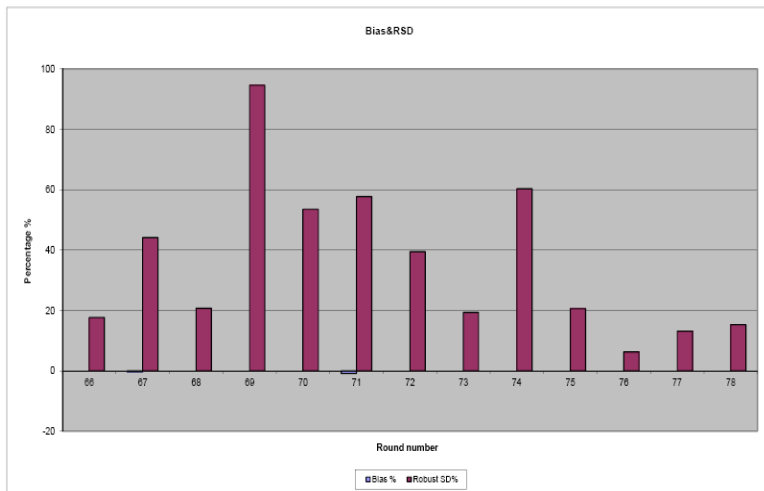
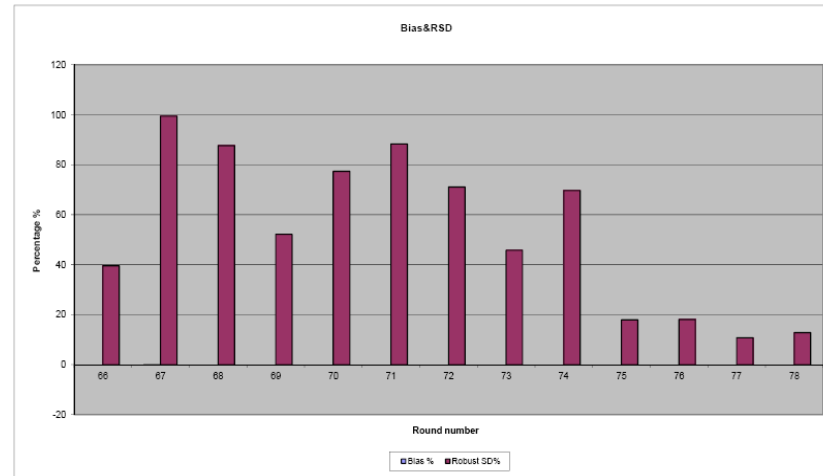
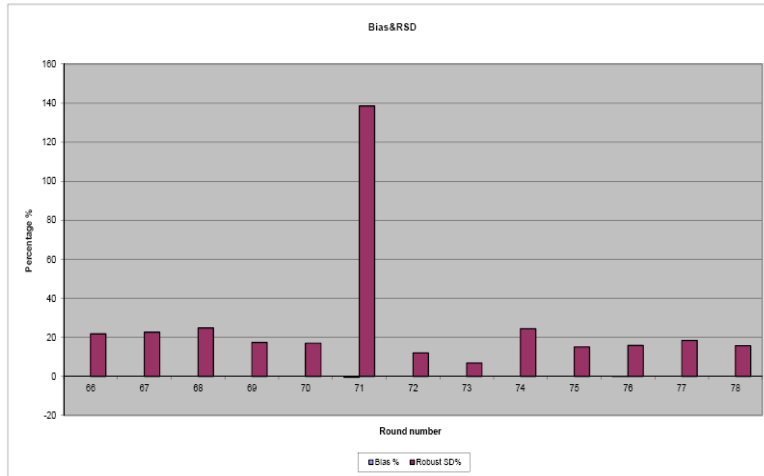
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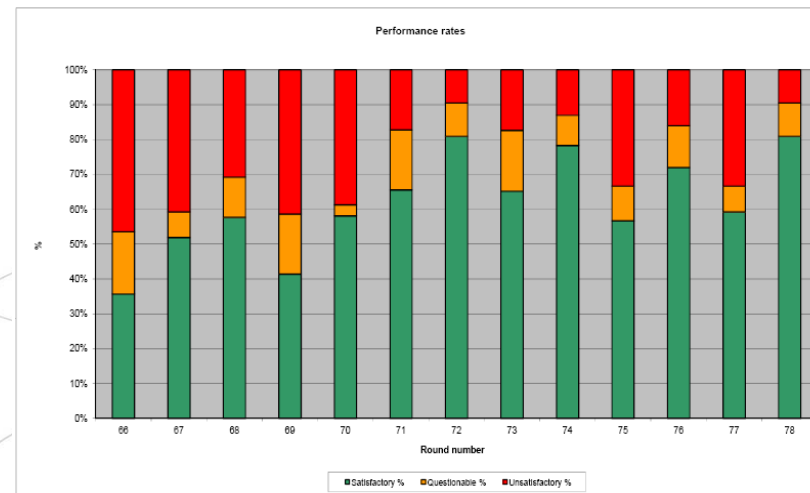
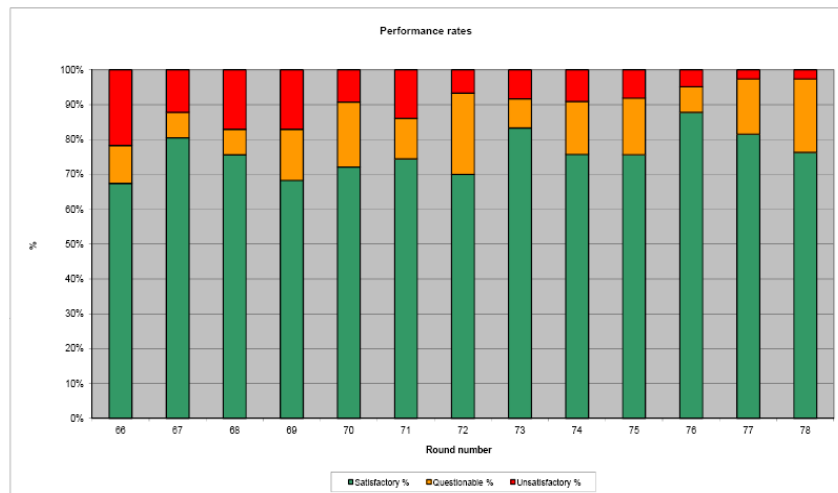
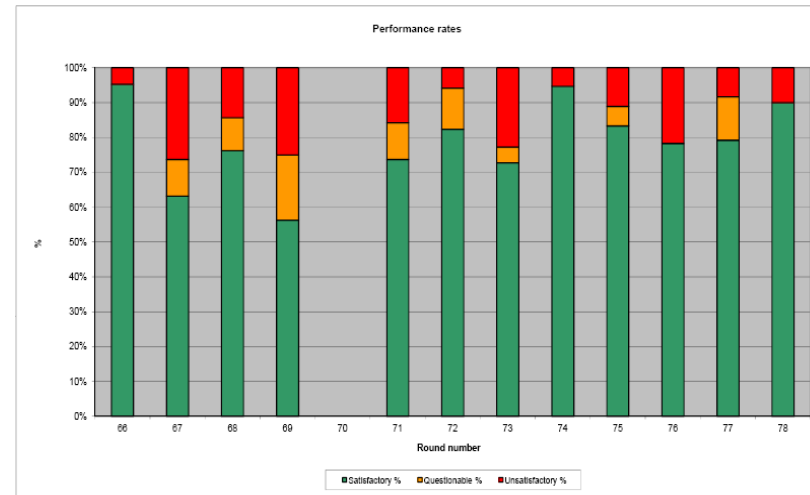
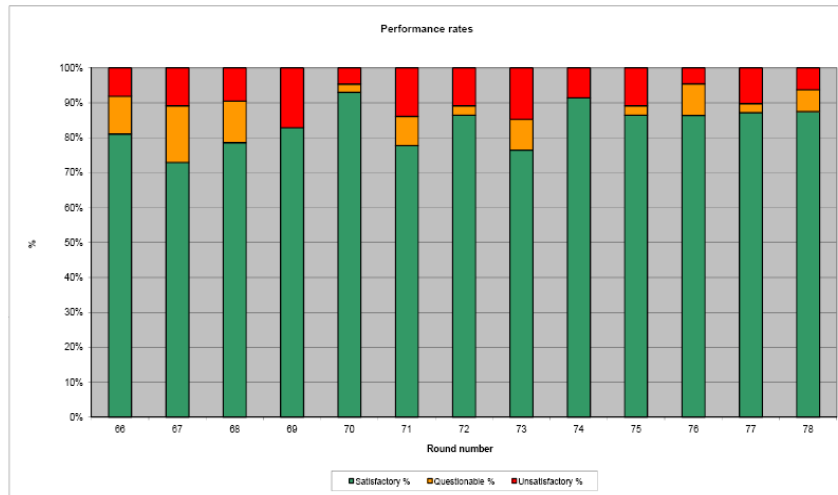
CONTEST: Sample B (CN, NH3, Thiocyanate, Soluble Boron)



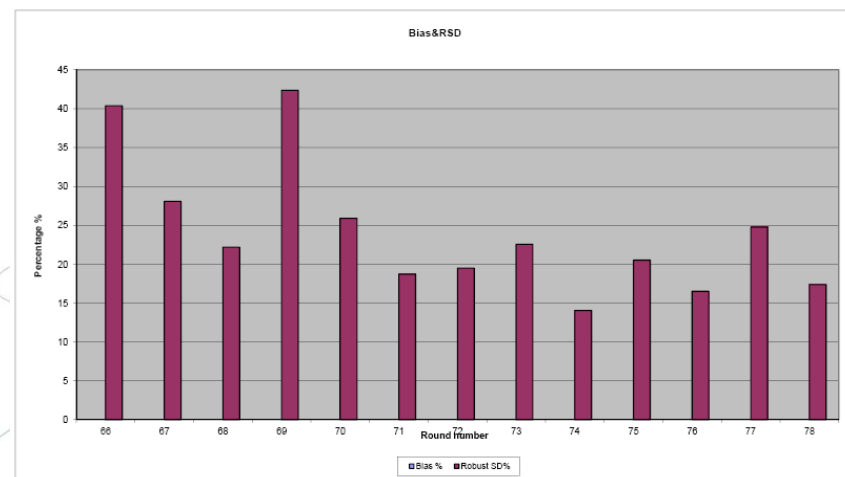
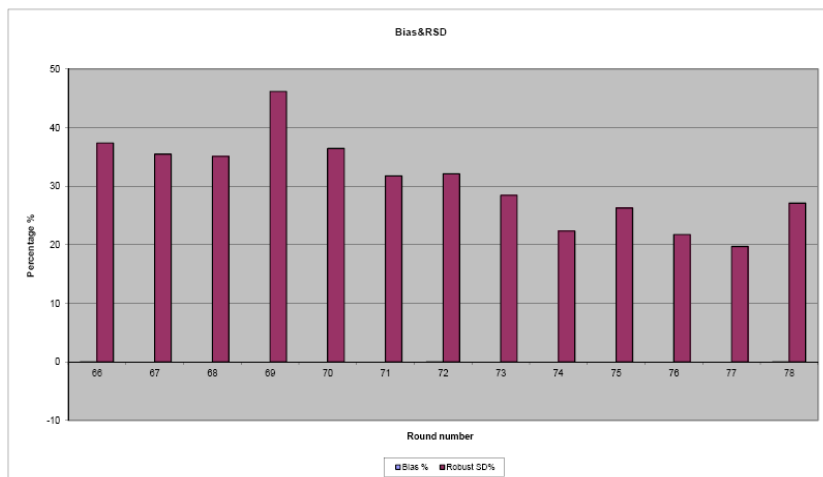
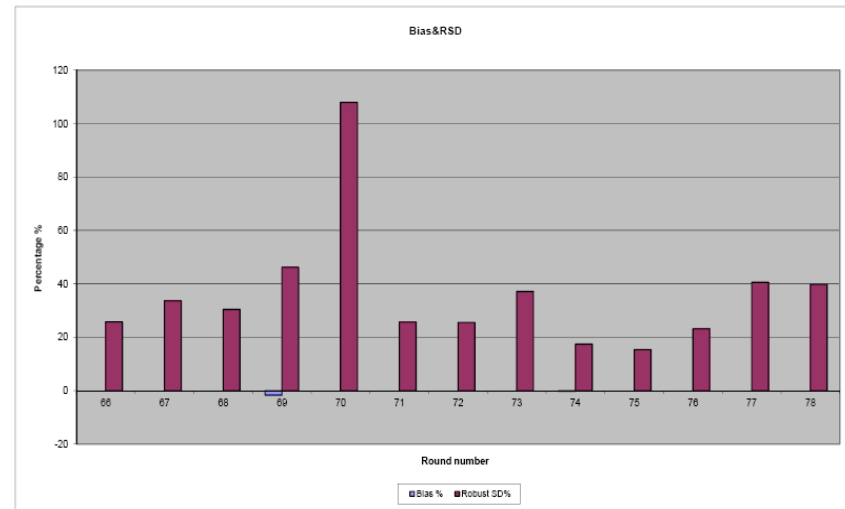
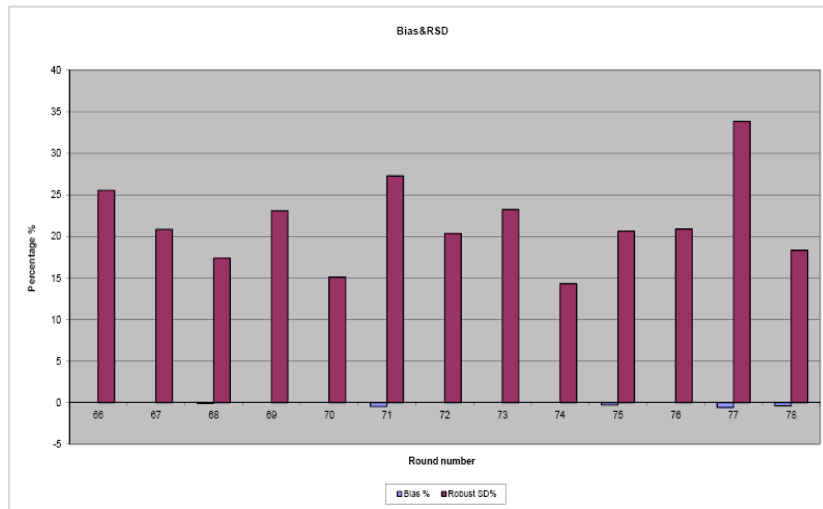
CONTEST: Sample B (CN, NH3, Thiocyanate, Soluble Boron)



CONTEST: Sample C (Anthracene, PCB 180, TPH, TOC)



CONTEST: Sample C (Anthracene, PCB 180, TPH, TOC)



CONTEST: Performance Summary



- CONTEST is a well established scheme
- Performance is stable for the majority of analytes
- Improvements have been observed (% satisfactory and %RSD) in analytes or analyte groups
 - Where the analysis is ‘difficult’, Sample A, Mercury etc.
 - Where samples with higher concentrations have been provided, Sample B, Ammonia and Thiocyanate.
 - In samples where LGC and the CONTEST advisory group have ‘worked with’ participants, Sample C, TOC and TPH.

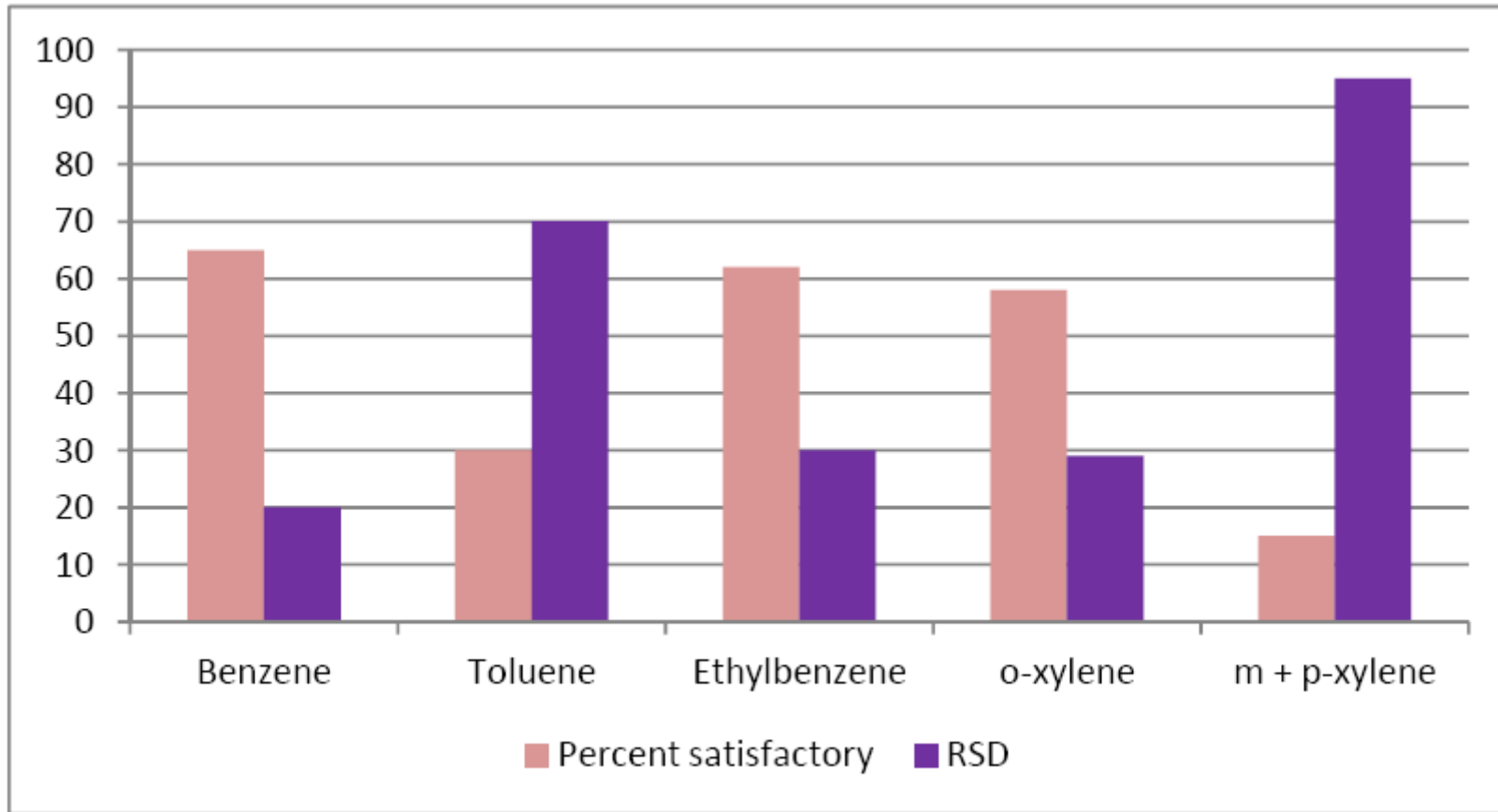
CONTEST: BTEX in soil

- Structure
 - 30g soil (dried and ground)
 - 2ml standard solution in methanol
- Preparation
 - 0.5ml of standard solution is added to 10g of the soil supplied.
- Analytes
 - Benzene, Toluene, Ethylbenzene, Xylene (ortho, meta + para)
- Results
 - Reported in mg analyte per Kg
- Statistics
 - AV = Formulation, SDPA = 20% of AV

CONTEST: BTEX in soil



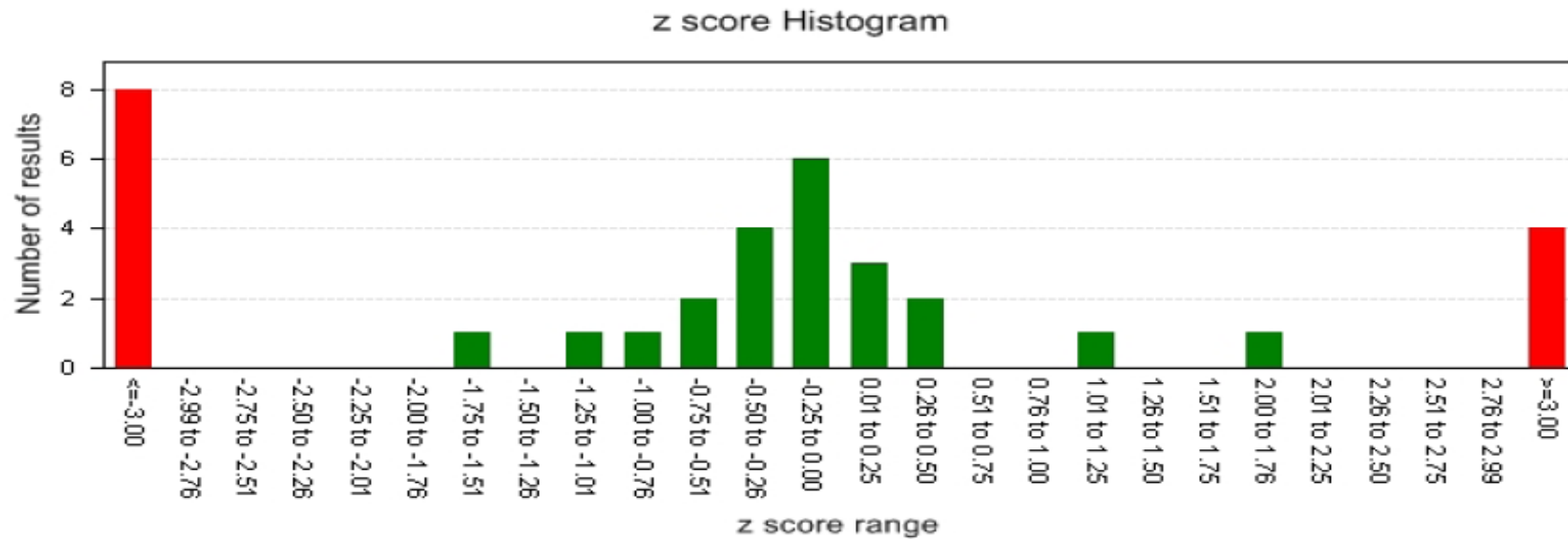
Excellence through measurement



CONTEST: BTEX (Benzene)



Excellence through measurement



Data Statistics

	Value
Number of Results	34
Number of Excluded Results	3
Mean	1.59 mg/kg
Median	1.93 mg/kg
Standard Deviation	0.91 mg/kg
Robust Standard Deviation	0.40 mg/kg
Result Range	0.02 to 3.45 mg/kg

Performance Statistics

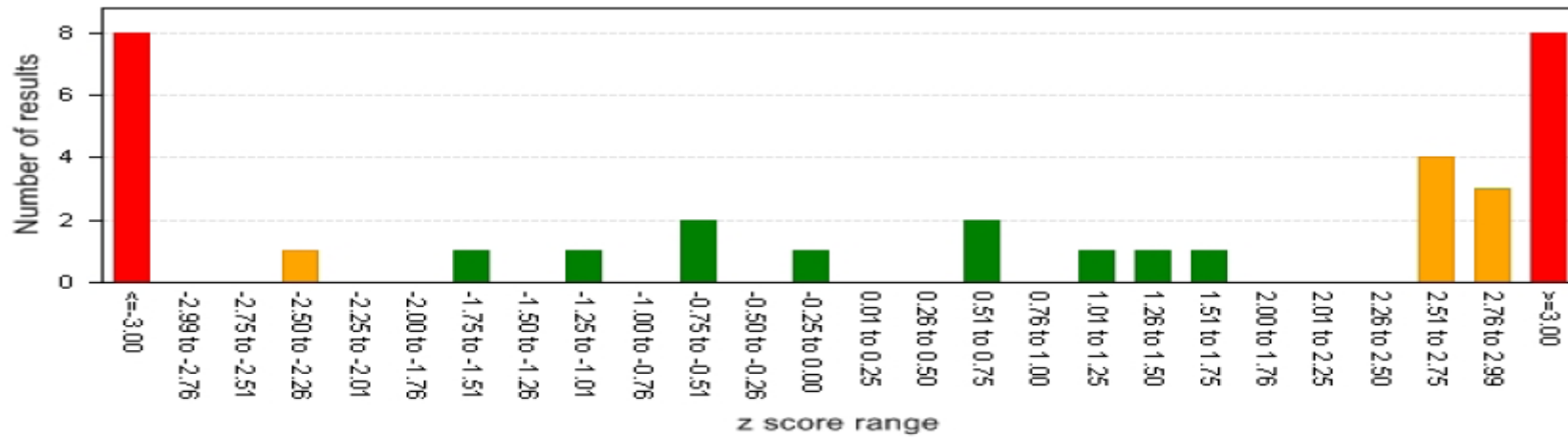
	Value
Assigned Value	2.08 mg/kg
Uncertainty of Assigned Value	0.01 mg/kg
SDPA	0.42 mg/kg
Satisfactory Range	1.24 to 2.92 mg/kg
Satisfactory z scores	64.7%
Questionable z scores	0.0%
Unsatisfactory z scores	35.3%

CONTEST: BTEX (Toluene)

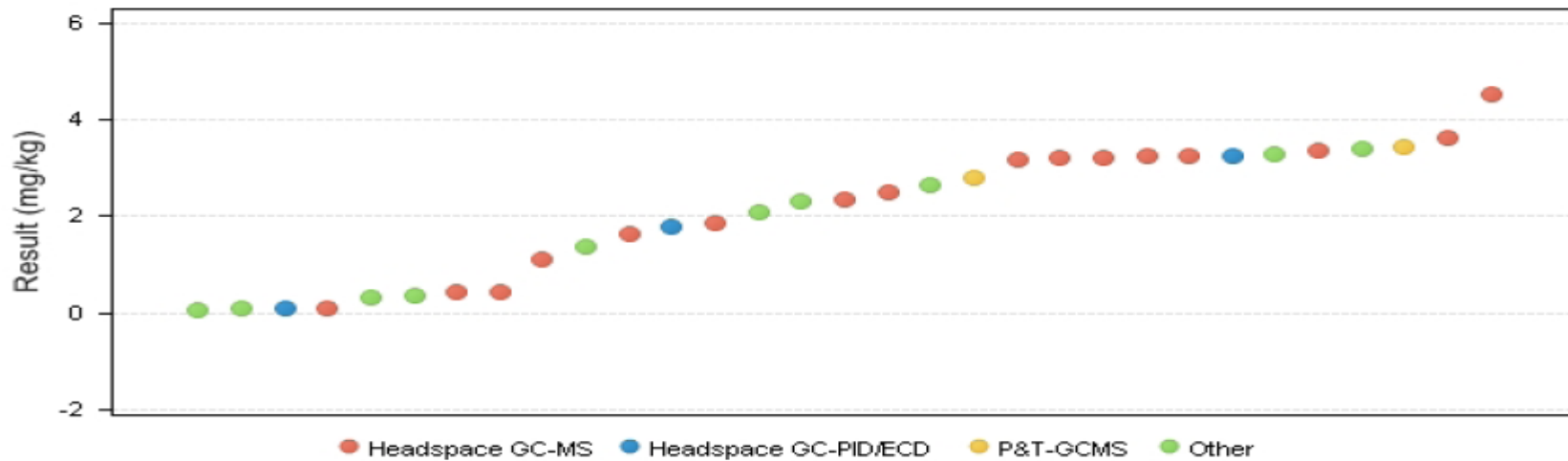


Excellence through measurement

z score Histogram



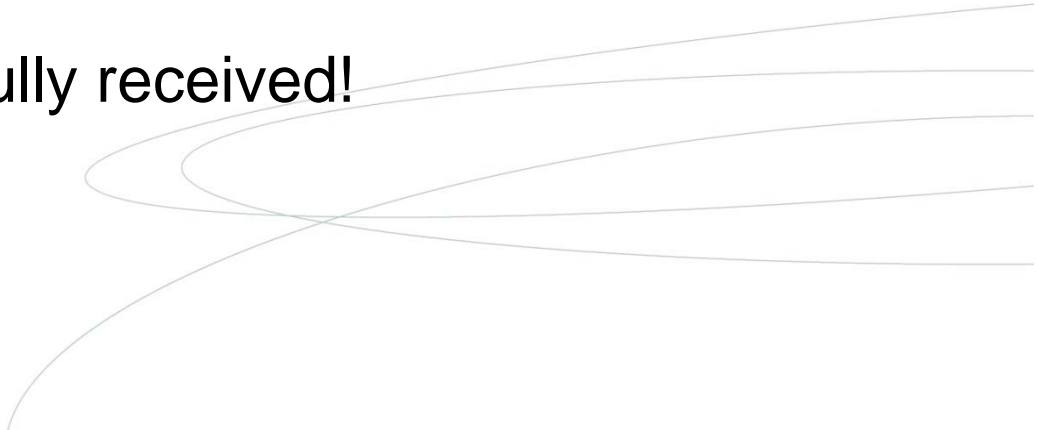
Distribution Graph



CONTEST: BTEX (Summary)

- Performance good for 3/5 analytes
 - Benzene, Ethylbenzene and o-xylene
- Poor performance for 2/5
 - Toluene and m+p-xylene
 - Low rate of satisfactory performance
 - Large spread of results
- Some participants did not calculate results correctly
- Soil may have contained background levels of some analytes
 - Toluene and m+p-xylene

CONTEST developments 2012/13

- BTEX in soils
 - Sample provided three times
 - Sample format similar to the 2011 trial sample
 - Similar material for low molecular weight TPH (C6-10)
 - WAC Leaching sample
 - 3 times per year
 - Evenly distributed rounds '1,3 and 5'
 - All suggestions gratefully received!
- 

Thank-you for listening



Any questions!