

Addressing the Difficulties of chemical testing

By Peter Hewitt

Lies, Damned Lies & Lab Testing

Introduction

- **The Beginning in Grays**
- **Lab accuracy, sample location and LOD**
- **MCERTS and CONTEST**
- **Stones and Extractants**
- **The Answer, BS10175 and Prof Ramsey?**
- **Split and Duplicate Sampling**
- **Pop Quiz Reveal**
- **Conclusion**

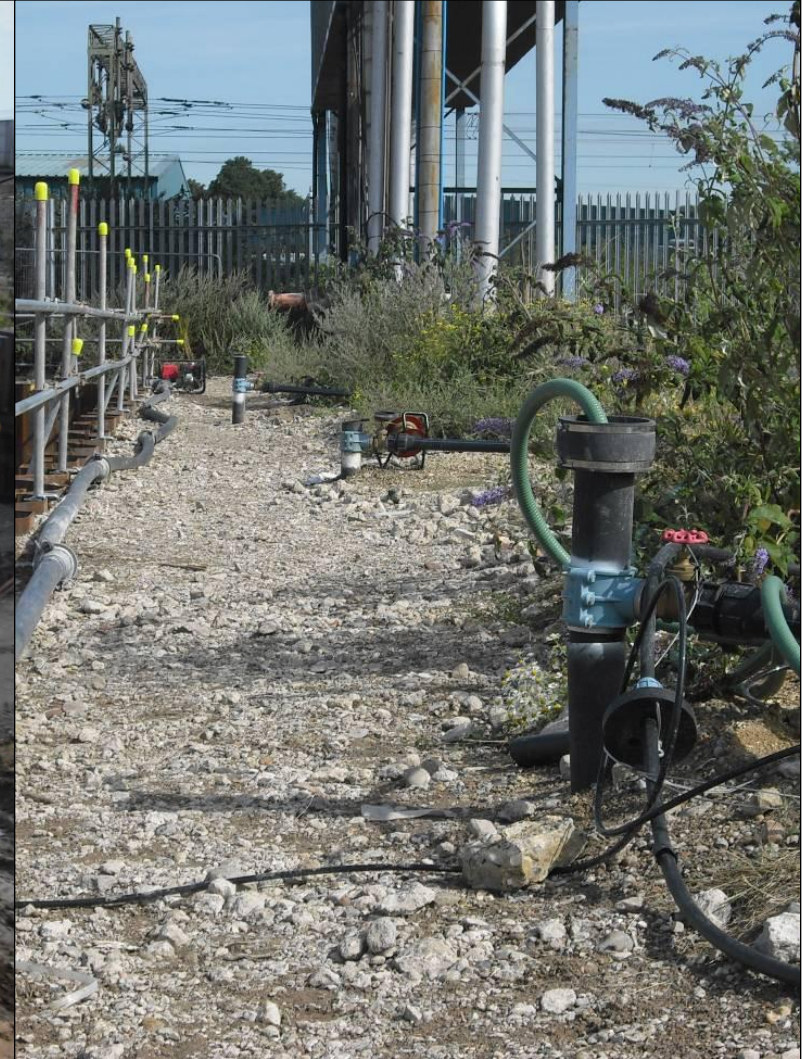
Pop Quiz - 1944



O'Rourke Yard - Grays



Grays - Remediation



Grays – Split Sampling



	Lab 1	Lab 2	Lab 3
TPH (mg/kg)	4,800	2,583	1,863

Grays – Sample Selection



	SP 1	SP 2	STK
TPH (mg/kg)	6,596	3,955	1,304

Limit of Detection

Heathrow T2A

Environment Agency Discharge Consent: COD 10mg/kg

Laboratory Testing Limit of Detection: 20mg/kg



What Does MCERTS Mean?



1. Performance targets
2. Selection and validation of methods
3. Sampling pre-treatment and preparation
4. Participation in proficiency scheme
5. Reporting of results and methods

Performance Standard for
Laboratories Undertaking
Chemical Testing of Soil

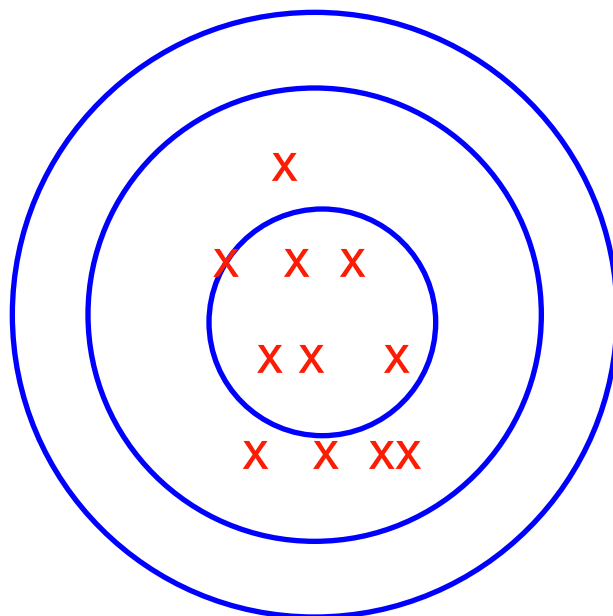
Environment Agency
March 2012
Version 4

- It does NOT certify the Lab only the test
- It does NOT define a test method
- It does NOT cover all testing

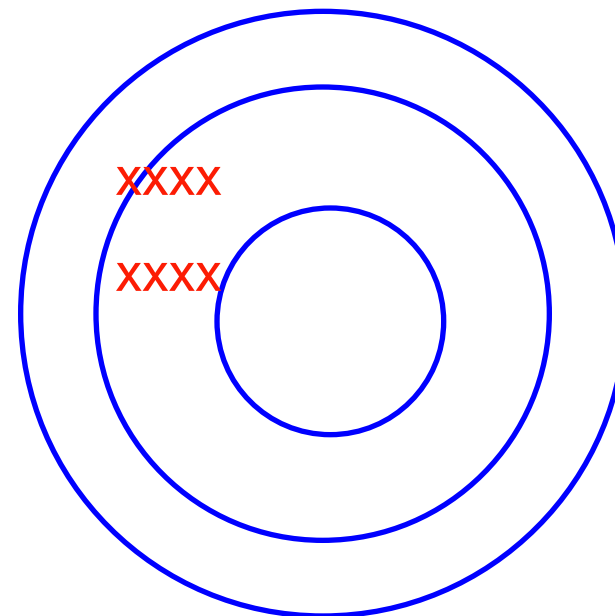


MCERTS Defined Limits

PRECISION



BIAS



Metals

7.5%

10%

Organometallics

15%

30%

Organics

10%

20%

Inorganics

15%

30%

Impact of Sample Prep

1. Sample selection (10 – 20g)

- Cone and quarter
- Wet/Dry split
- Crush

2. Grading

- 10mm
- 2mm
- All

3. Extraction (Solvent)

- Method
- Solvent used

Extraction (Solvent)

- Hexane/Acetone
- DCM/Methanol
- DCM/Pentane
- Cyclohexane

Proficiency Scheme - CONTEST

- Operated by the LGC
- Based on testing Standardised 200um sample
- Not all tests are included in CONTEST rounds
- Laboratories do not have to participate in all rounds
- Results based on Z scores
- $Z = (X_1 - X_m) / \text{Horwitz}_{sd}$ (X_1 = Reported, X_m = Median Result)
 - $Z < 2$ Satisfactory
 - $2 < Z < 3$ Questionable
 - $3 < Z$ Unsatisfactory

Proficiency Scheme - CONTEST

Total PAHs in prepared soil



LAB.	EAGLE	CONTEST
1	74.1	88.9
2	71.6	131.4
3	38.0	75.9
4	61.3	84.1
5	88.1	15.3
6	75.1	68.1
7	63.4	170
8	70.3	79.6
9	81.5	980*
10		61.2
11		53.9
12		103.9
13		77.2
14		191
15		114
16		74.2
17		95.9
18		91
19		54.9
20		113
MEAN	73.1	136.2
RANGE	61.3-88.1	15.3-980

Proficiency Scheme - CONTEST

Sample: 21 - Group E Soils for Waste Water Acceptance Criteria

Analyte: Selenium

Lab ID	Method	Result (mg/kg)	z' score*
CN0002	ICP-MS	0.1580	-1.06
CN0005	ICP-OES	0.2000	-0.64
CN0020	ICP-OES	0.6200	3.51
CN0035	ICP-MS	2.0300	17.44
CN0036	ICP-MS	6.4200	60.82
CN0057	ICP-MS	14.0000	135.73
CN0062	ICP-MS	0.6216	3.52
CN0101	Hydride gen/cold vapour	0.2400	-0.25
CN0109	ICP-MS	9.1088	87.40
CN0117	ICP-OES	<0.0500	
CN0133	ICP-OES	0.2000	-0.64
CN0148	ICP-MS	8.3370	79.77
CN0174	ICP-MS	0.2900	0.25
CN0175	Hydride gen/cold vapour	0.2100	-0.54
CN0263	ICP-OES	0.7630	4.92
CN0265	ICP-MS	0.3310	0.65
CN0269	ICP-MS	0.2400	-0.25
CN0276	ICP-OES	0.2200	-0.44
CN0280	ICP-MS	0.4990	2.31
CN0291	ICP-OES	<0.6000	
CN0292	ICP-OES	<1.0000	
CN0817	ICP-OES	0.2983	0.33

8 Results Excluded

LOD <1.0 to <0.05

Inductively Coupled
Plasma

Mass Spectrometer
Optical Emission Sp

Proficiency Scheme - CONTEST

Performance Statistics

	Value
Assigned Value	0.265 mg/kg
Uncertainty of Assigned Value	0.032 mg/kg
SDPA	0.096 mg/kg
Satisfactory Range	0.073 to 0.457 mg/kg
Satisfactory z' scores	52.6%
Questionable z' scores	5.3%
Unsatisfactory z' scores	42.1%

WAC Limit 0.1mg/kg

Whats the answer – BS10175?

BS 10175:2011+A1:2013

- App D – The assessment and control of sample uncertainty
- (Informative)

RAMSEY, M. H., and ELLISON, S.L.R. (eds.) EURACHEM/EUROLAB/CITAC/NORDTEST/AMC. *Guide: Measurement uncertainty arising from sampling: A guide to methods and approaches*. Prague: EURACHEM, 2007.

- The use of split and duplicate samples



Investigation of potentially contaminated sites – Code of practice

bsi.

...making excellence a habit.™

LAING O'ROURKE

Explore Manufacturing



Explore Manufacturing

	Zinc	Spec PAH	Aliphatic C21 – C35
Median	237	6.65	13.15
Std dev	179	82	90
Range	77 – 670	1.48 - 263	9.7 - 246
Duplicate Error	24%	52%	30%
Split Error	19%	39%	20%

Explore Manufacturing



Pop Quiz – ?



Pop Quiz – Heathrow T2A



Heathrow T2A



Heathrow T2A

Water Sample Well 5, 21st May 2010



09:42



10:14



10.29



10:46

Heathrow T2A

SAL Reference: 200361

Customer Reference: Heathrow

Water

Analysed as Water

Miscellaneous

SAL Reference					200361 001	200361 002	200361 003	200361 004	200361 005
Customer Sample Reference					T2A Well 1	T2A Well 2	T2A East Siltbuster	T2A West Siltbuster	T2A Well 5
Determinand	Method	Test Sample	LOD	Units					
Ferric Iron (Fe III)	T85	AR	0.01	mg/l	12	0.78	2.0	2.0	3.0
Ferrous Iron	T4	AR	0.01	mg/l	0.80	0.15	15	21	31
Iron	T154	AR	0.05	mg/l	13	0.93	17	23	34

- Unless otherwise specified, water samples are filtered
- Check for time dependant changes
- Iron is not on standard list of Contaminants
- Match testing to needs; Contam Land leaves gaps for Waste and Discharge

Heathrow T2A



London Gateway Port



Conclusions

- **Talk to your laboratory**
- **Re-take Chemistry and Maths A Level**
- **Understand testing method and limits**
- **Embrace uncertainty**
- **Check MCERTS coverage**
- **Check CONTEST Z scores**
- **Use BS10175**
- **Match the testing to the need**

THANK YOU