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Weighing Proficiency Testing Scheme for the STA, Round 2 Final Report, 2nd Issue

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Weighing Proficiency Testing Scheme for the STA, Round 2
Final Report, 2nd Issue

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ABSTRACT

This report presents the results of the second round of the particulate weighing proficiency testing scheme operated by NPL for the Source Testing Association completed in November 2008.

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by

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1. INTRODUCTION

This report describes the results of the second round of a weighing measurement proficiency-testing scheme carried out by the National Physical Laboratory for the Source Testing Association (STA).

The weighing proficiency testing (PT) scheme provides a way of assessing the performance of laboratories by a series of regular inter-laboratory comparisons. The test samples consisted of foil shims, with known masses provided by traceable measurements carried out by NPL, and solutions containing known mass of dissolved salt, simulating a probe washing solution. The results of each participant's analyses were compared to the assigned values. The set of results have been reported anonymously, and in addition each participant has been made aware of their own results. In this way participants are able to assess their performance in relation to other laboratories.

2. PT SCHEME DESCRIPTION

Thirty-seven STA member companies took part in the second round of the scheme, which involved the round robin measurement of shims and simulated probe washing solutions.

The PT scheme assesses the measurement by the participants, of the masses of metal foil shims, and the masses of the residual salt following evaporation of the solutions. The test samples each have a known, assigned value, determined by NPL. In this PT scheme each participant received a separate test sample.

The shims were individually weighed by NPL, after being cleaned using alcohol and then heated in an oven in accordance with the procedure in BS EN 13284-1.

The washing solution samples were decanted by pipette from a bulk stock solution. The bulk solution contained a known mass of salt (NaCl) in a measured volume of purified water. The solutions delivered to the participants each contained a nominal mass of salt of 10 mg. The shims had nominal masses in the range 122 mg to 158 mg.

The participants were not told the masses of the shims or the salt content of the solution. Each participant was required to weigh their shim using a weighing procedure in accordance with BS EN13284-1. The shims were dried in a drying oven for at least one hour, at a minimum of 180 °C, and then cooled down to ambient temperature in a desiccator located in the weighing room for at least 4 hours. The shims were then weighed and the weight recorded after 1, 2 and 3 minutes and the result extrapolated to zero time and reported.

The washing solution was split by the participants into three equal parts. These were then evaporated following their standard methodology and weighed, again in accordance with BS

EN 13284-1. The results of the three washing solution weighings were reported. The sum of these three weights should equal the 10.6mg known to be in the solutions provided to the participants.

The results of these analyses were returned to the STA and NPL.

The results of each analysis were expressed as percentage differences from the true value. This allows a comparison to be made between participants with different shim weights. The results are presented in graphical form in Figure 1 and 2, and a summary of the results is also given in Table 1 and 2. The participants are listed in random order.

There are a number of ways in which the results of PT schemes can be interpreted. The most straightforward technique is to examine the percentage differences of the reported results from the true value, as has been applied above to this PT scheme. It is then left to the participants to gauge how well they have performed.

A more refined interpretation of PT scheme results involves the calculation of a performance score for each result. This is usually based on comparing the results achieved against an assigned target standard deviation, σ. The simplest form of this is the 'z score'. This is calculated by dividing the deviation of each result from the true value by σ , see Equation 1.

$$z = \frac{x - T}{\sigma} \qquad \text{Equation 1}$$

where:

	z	z score
	x	value obtained by participant
	T	true value for test sample
	σ	assigned value for standard deviation

This provides a z score for each result, which can be compared with other z scores either by other participants or by the same participant on different rounds of the scheme. If a suitable value of σ is chosen then the z score also provides a method of deciding decision limits for the PT scheme. In general, if all results are normally distributed about the true value of the test sample and a reasonable value of σ has been chosen, then few (< 5 %) of the z scores should lie outside ± 2. z scores lying outside ± 3 would be strongly indicative of a true bias in the reported value, rather than random uncertainty. From this it is possible to apply a classification as follows:

$\|z\| \leq 2$	satisfactory
$2 > \|z\| < 3$	questionable
$\|z\| \geq 3$	unsatisfactory

These limits allow each participant to judge their own performance and can be used to indicate potential problems. The target standard deviation is usually taken to be a value, which is fit for purpose for the measurements being made. The value of σ used for the shim element of the scheme has been selected as 0.1 mg, this has been chosen to be 10% of a 1 mg particulate loading, and is also close to the 0.05 mg standard deviation observed in the results. The value of σ used for the washings element of the scheme has been selected as 2 mg. This relatively lenient target standard deviation has been chosen because of the additional complexity of required to measure a washing solution.

Participants who attain z scores of 2 or higher should investigate the cause of the performance with an aim to improving their performance in subsequent rounds. Those with z scores of 3 or higher should put in place a documented mechanism to correct any issues identified as soon as possible.

3. RESULTS

The results for the shim weighings are presented in Table 1.

Company I/D	Shim ID	Initial Shim Weight (mg)	Reported Shim Weight (mg)	Deviation of Shim Weight (mg)
1	14	124.36	124.35	-0.01
2	3	122.21	122.28	0.07
3	13	130.86	130.86	0.00
4	15	124.95	124.81	-0.14
5	8	122.59	122.59	0.00
6	32	126.55	126.59	0.04
7	33	127.58	127.51	-0.07
8	20	139.39	139.41	0.02
9	27	132.48	132.56	0.08
10	5	148.66	148.60	-0.06
11	11	128.26	128.27	0.01
12	25	134.84	135.00	0.16
13	9	132.95	132.98	0.03
14	37	131.14	131.09	-0.05
15	1	146.06	146.07	0.01
16	23	133.08	133.00	-0.08
17	16	126.27	126.28	0.01
18	28	131.20	131.44	0.24
19	31	128.90	128.90	0.00
20	6	140.64	140.60	-0.04
21	18	149.16	149.20	0.04
22	26	135.57	135.61	0.04
23	36	126.14	126.24	0.10
24	29	124.60	124.70	0.10
25	34	134.67	134.66	-0.01
26	2	158.04	157.99	-0.05
27	22	130.65	130.55	-0.10
28	30	124.75	124.75	0.00
29	7	142.57	142.50	-0.07
30	12	124.96	124.92	-0.04
31	21	124.67	124.80	0.13
32	4	144.14	144.17	0.03
33	19	129.02	128.93	-0.09

34	17	136.56	136.61	0.05
35	24	126.06	126.20	0.14
36	10	153.64	153.73	0.09
37	37	131.14	131.09	-0.05

Table 1. Difference, in mg, between reported results and NPL assigned value

The agreement with the assigned absolute shim weights is good. However, a comparison has to be made with the typical masses of particulate measured on filters, which is of the order of 1- 10 mg. The standard deviation of the differences is 0.05 mg, this provides an indication of the variability of weighing across the participants. The average difference in milligrams is 0.06 mg, which shows very good agreement with the known weights and no significant bias in the results.

The z score results for the shim measurements are given in Table 2.

Company I/D	Shim ID	% Difference from assigned value	Z Score σ = 0.1mg
1	14	-0.01	0.13
2	3	0.06	0.73
3	13	0.00	0.03
4	15	-0.11	1.37
5	8	0.00	0.03
6	32	0.03	0.43
7	33	-0.05	0.70
8	20	0.02	0.23
9	27	0.06	0.80
10	5	-0.04	0.57
11	11	0.01	0.07
12	25	0.12	1.60
13	9	0.02	0.27
14	37	-0.04	0.50
15	1	0.01	0.13
16	23	-0.06	0.77
17	16	0.01	0.07
18	28	0.18	2.37
19	31	0.00	0.03
20	6	-0.03	0.37
21	18	0.03	0.43
22	26	0.03	0.37
23	36	0.08	1.00
24	29	0.08	0.97
25	34	-0.01	0.10
26	2	-0.03	0.53
27	22	-0.08	1.03

28	30	0.00	0.03
29	7	-0.05	0.70
30	12	-0.03	0.40
31	21	0.11	1.33
32	4	0.02	0.29
33	19	-0.07	0.90
34	17	0.03	0.47
35	24	0.11	1.37
36	10	0.06	0.90
37	37	-0.04	0.50

Table 2. Shim results, percentage difference and z scores

The z score results for the shims are good, with only one participant with a z-score greater than 2. Figures 1 and 2 show these results graphically.

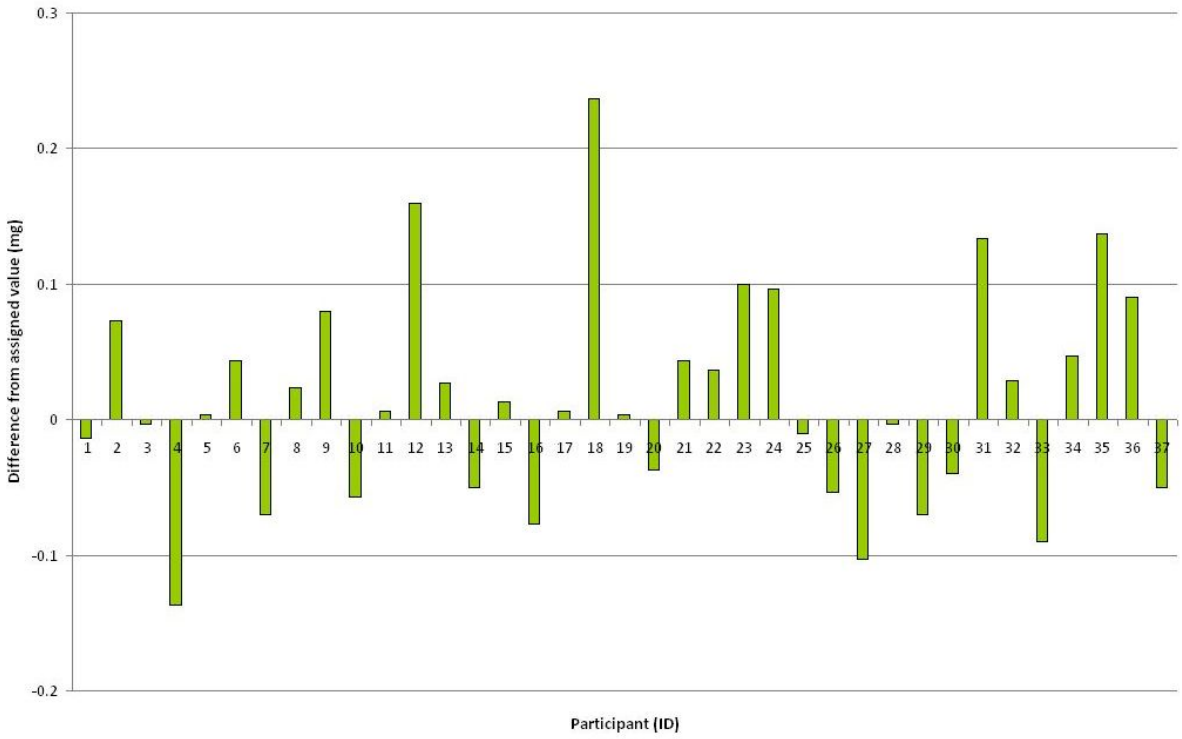

Figure 1. Difference in shim weights from assigned values

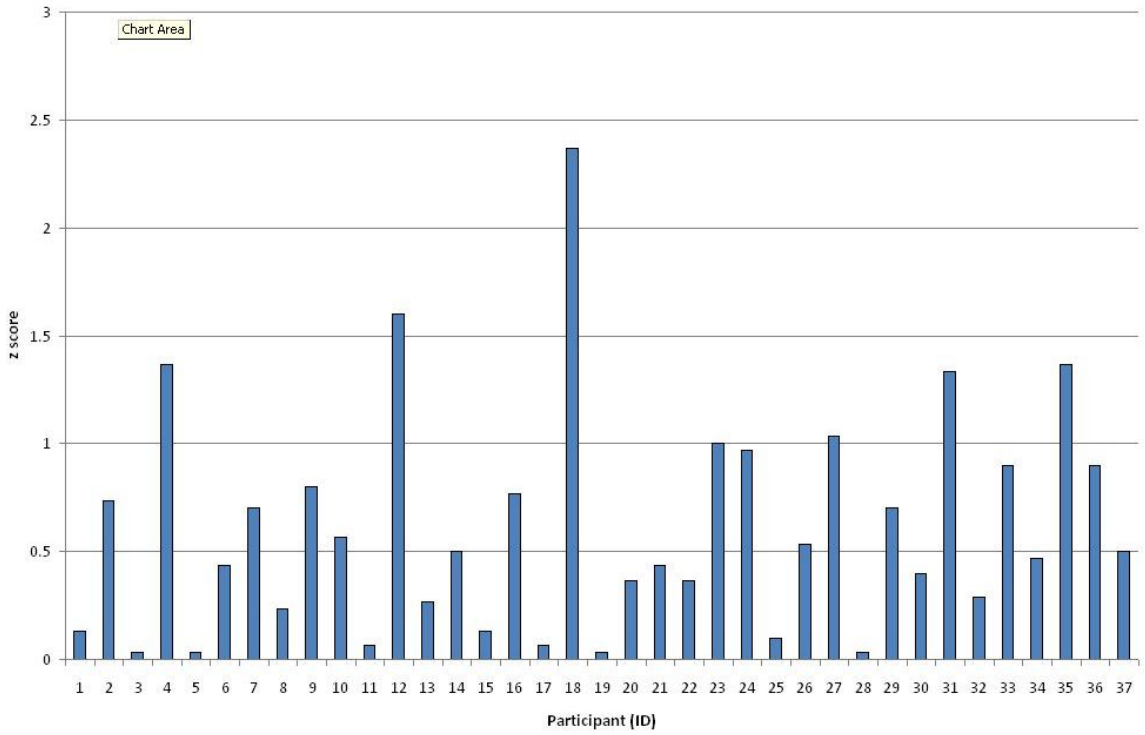

Figure 2. z scores for shim weighing results, σ = 0.1mg

The results for the solution evaporation and weighing are given in Table 3. This gives the results from weighing the residuals from the 3 portions of the solution and the total mass of residue from all three, which should be equal to the 10.6 mg salt present in the provided samples.

Company I/D	Mass salt assigned value (mg)	Wash one (mg)	Wash two (mg)	Wash Three (mg)	Total Wash Weights (mg)	Deviation from set value (mg)
1	10.60	4.14	3.65	3.49	11.28	0.68
2	10.60	3.29	3.16	4.22	10.67	0.07
3	10.60	3.32	2.99	3.83	10.14	-0.46
4	10.60	3.40	3.50	3.70	10.60	0.00
5	10.60	0.00	0.00	14.90	14.90	4.30
6	10.60	5.00	5.00	5.00	15.00	4.40
7	10.60	3.00	2.69	2.49	8.18	-2.42
8	10.60	3.88	3.73	3.49	11.10	0.50
9	10.60	3.40	3.40	3.50	10.30	-0.30
10	10.60	4.43	4.47	4.76	13.66	3.06
11	10.60	3.60	3.90	3.80	11.30	0.70
12	10.60	3.30	2.30	4.00	9.60	-1.00
13	10.60	3.92	3.43	3.94	11.29	0.69
14	10.60	3.60	3.80	3.70	11.10	0.50
15	10.60	1.87	2.92	0.11	4.90	-5.70

16	10.60	3.30	3.20	4.00	10.50	-0.10
17	10.60	3.70	3.60	3.60	10.90	0.30
18	10.60	4.00	3.60	3.50	11.10	0.50
19	10.60	4.10	5.10	3.90	13.10	2.50
20	10.60	3.42	3.66	3.69	10.77	0.17
21	10.60	3.60	3.60	3.20	10.40	-0.20
22	10.60	1.52	1.43	1.90	4.85	-5.75
23	10.60	3.53	4.07	3.83	11.43	0.83
24	10.60	0.00	0.00	8.90	8.90	-1.70
25	10.60	3.08	2.84	3.65	9.57	-1.03
26	10.60	3.37	3.35	3.61	10.33	-0.27
27	10.60	3.47	3.39	3.37	10.23	-0.37
28	10.60	5.30	5.40	10.90	21.60	11.00
29	10.60	4.60	3.40	3.90	11.90	1.30
30	10.60	3.74	3.65	3.75	11.14	0.54
31	10.60	3.40	4.30	4.20	11.90	1.30
32	10.60	2.41	3.46	1.83	7.69	-2.91
33	10.60	0.00	0.00	6.45	6.45	-4.15
34	10.60	3.90	4.10	4.10	12.10	1.50
35	10.60	3.30	3.60	3.50	10.40	-0.20
36	10.60	3.30	3.03	3.93	10.26	-0.34
37	10.60	3.60	3.80	3.70	11.10	0.50

Table 3. Difference, in mg, between reported results and NPL assigned value

Table 4 presents the washing solution results as percentage difference from the assigned value and as a z-score, with a target standard deviation of 2 mg.

Company I/D	% Difference from assigned value	Z Score σ = 2mg
1	6.42	0.34
2	0.66	0.04
3	-4.34	0.23
4	0.00	0.00
5	40.57	2.15
6	41.51	2.20
7	-22.83	1.21
8	4.72	0.25
9	-2.83	0.15
10	28.87	1.53
11	6.60	0.35
12	-9.43	0.50
13	6.51	0.35
14	4.72	0.25

15	-53.77	2.85
16	-0.94	0.05
17	2.83	0.15
18	4.72	0.25
19	23.58	1.25
20	1.60	0.09
21	-1.89	0.10
22	-54.25	2.88
23	7.83	0.42
24	-16.04	0.85
25	-9.72	0.52
26	-2.55	0.14
27	-3.49	0.19
28	103.77	5.50
29	12.26	0.65
30	5.09	0.27
31	12.26	0.65
32	-27.42	1.45
33	-39.15	2.08
34	14.15	0.75
35	-1.89	0.10
36	-3.21	0.17
37	4.72	0.25

Table 4. Washing solutions, percentage differences and z scores

Figures 3 and 4 present the percentage difference and z-scores for the washing solution results.

The z-score for the solution evaporation and weighing results has been calculated using a target standard deviation, σ, of 2 mg. Despite this relatively conservative target (approximately equivalent to 20% of the assigned value of 10.6 mg), the results are variable, with one participant gaining a z-score in excess of 3. The standard deviation of the differences from the expected weights was 2.25 mg, which is close to the assigned 2 mg standard deviation. It is also interesting to note there is significant variability between the weights of the three split washings from some participants, indicating possible sources of variation due to contamination or material loss during sample handling. It also appears some participants did not split the solutions or did not report the individual results (5, 24 and 33).

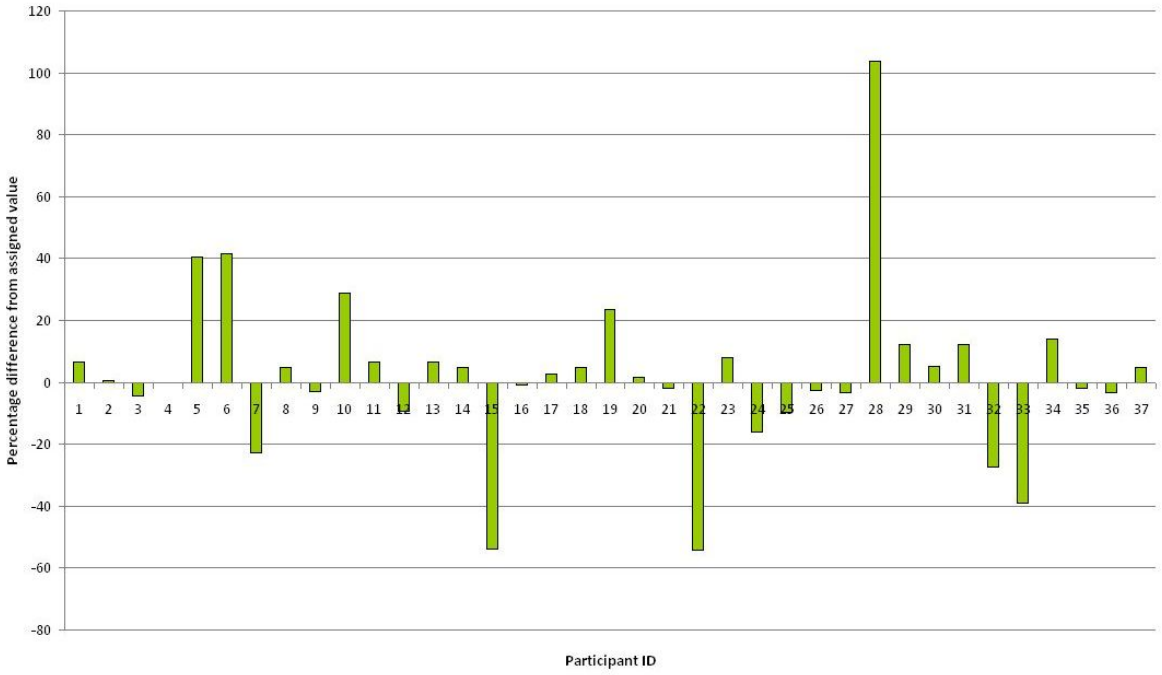

Figure 3. Percentage Difference in washing solution residual weight from assigned values

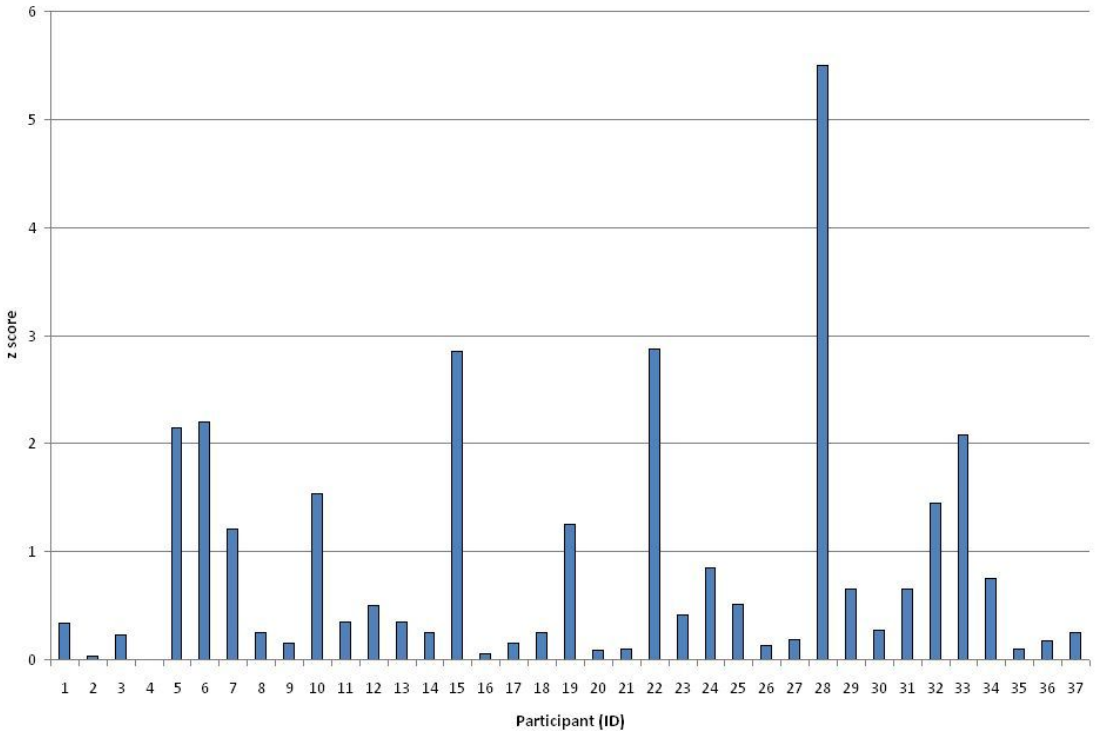

Figure 4. Washing Solution residue z score, σ = 2mg

4. ROUND COMPARISON

This is the second round of the weighing proficiency testing scheme. Further rounds of the scheme will enable us to look for trends in results and enable participants to judge their performance year on year. The z scores from round one and two for shims and washings are shown in Figures 5 and 6. For the washings element of the scheme 68% of participants achieved an improved z score from round 1. However only 54% showed an improvement in z score for the shim weighing.

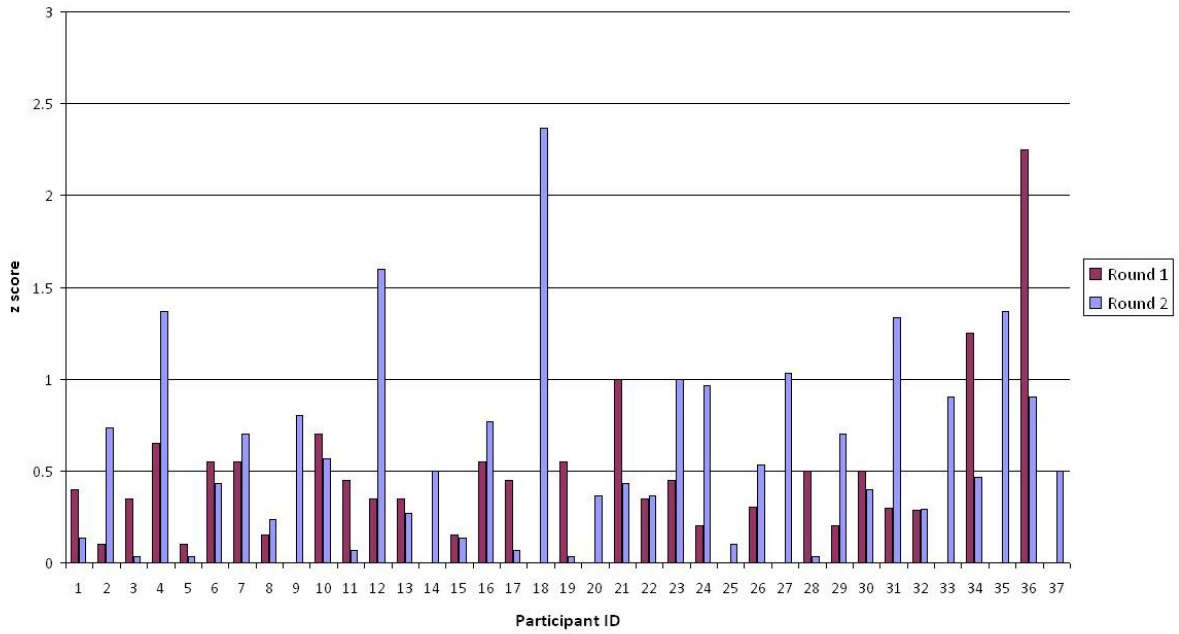

Figure 5. Round comparison of shim z scores

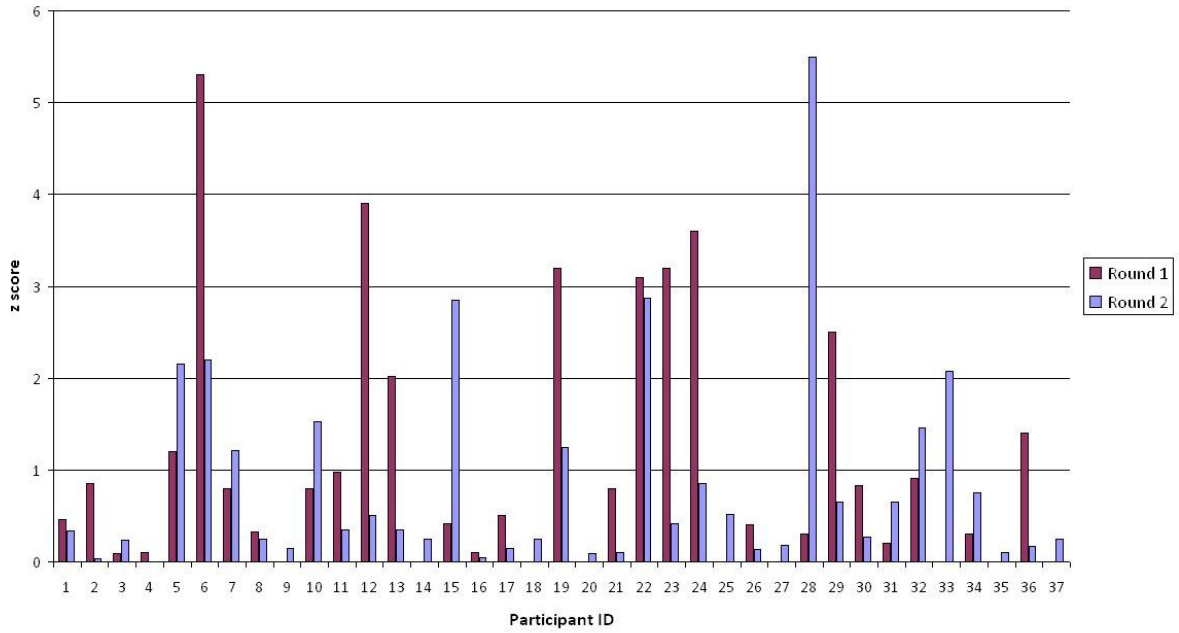

Figure 6. Round comparison of washings z scores

5. CONCLUSIONS

The results from the shim measurements are generally encouraging and indicate that most labs have their balances under calibration. 54% of labs achieved a lower shim z score this round compared to last. Participants with higher z scores are encouraged to investigate possible causes.

The results from the solution evaporation and weighing are much more encouraging and show significant improvement from the last round. All labs bar one achieved a z score less than 3 but there is still room for improvement with six labs with z scores in excess of the $0 \leq 2$ acceptable range. There is still a lot of variation in the results with an average difference from the assigned value of 1.68 mg and standard deviation of 2.25 mg.

6. ACKNOWLEDGEMENTS.

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