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A ROUND-ROBIN TO MEASURE THE DIRECT PIEZOELECTRIC COEFFICIENT USING THE BERLINCOURT METHOD

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A Round-Robin to Measure the Direct Piezoelectric Coefficient using the Berlincourt Method

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Abstract

An international round-robin exercise to measure the direct piezoelectric coefficient (d_{33}) using the Berlincourt method has taken place under the auspices of the Versailles Project on Advanced Materials And Standards (VAMAS). Three material compositions, representing both "soft" and "hard" piezoceramics, at two different nominal thicknesses, 1mm and 10mm, were distributed to the participating laboratories. Each laboratory received a separate set of specimens accompanied by a set of instructions specifying the measurement requirements and a list of controls and precautions, to be followed as closely as possible, and a report template for the results. All specimens were supplied poled and electroded, and were checked for outliers before being sent to participants.

The results obtained using different equipment configurations and different test parameters from those recommended showed large inter-laboratory variations; however the measurements made by following the recommendations supplied with the samples agreed well with each other. Data on the repeatability and reproducibility of the technique are presented.

Conclusions for the measurement procedure are: i) using current variable practices results in very poor inter-laboratory reproducibility ~15%; ii) using current variable practices produces good repeatability <5%; iii) following a standard procedure produces excellent repeatability <1%; iv) following a standard procedure results in very good inter-laboratory reproducibility <3%; v) soft, thin materials are a notable exception with repeatability ~4% and reproducibility ~10%, but this issue has long been recognised for these materials.

1.   Introduction

A round-robin to measure the direct piezoelectric coefficient using the Berlincourt method has been carried out under the auspices of the Versailles Project on Advanced Materials and Standards (VAMAS). The round robin is an activity of the VAMAS Technical Working Area 24 which is concerned with electroceramic performance related properties. Currently there are no standard methods for measuring the direct piezoelectric coefficient, d_{33}.

Previous investigations [1] have shown the Berlincourt d_{33} direct measurement method, shown schematically in Figure 1, to be affected by a number of factors:

- AC measuring force,
- AC frequency,
- static pre-load,
- sample geometry,
- contact point geometry,
- ambient conditions,
- time dependent relaxation.

However, this technique has no associated standard and without specific controls on many of these parameters, there is expected to be large variability in any quoted d_{33} values.
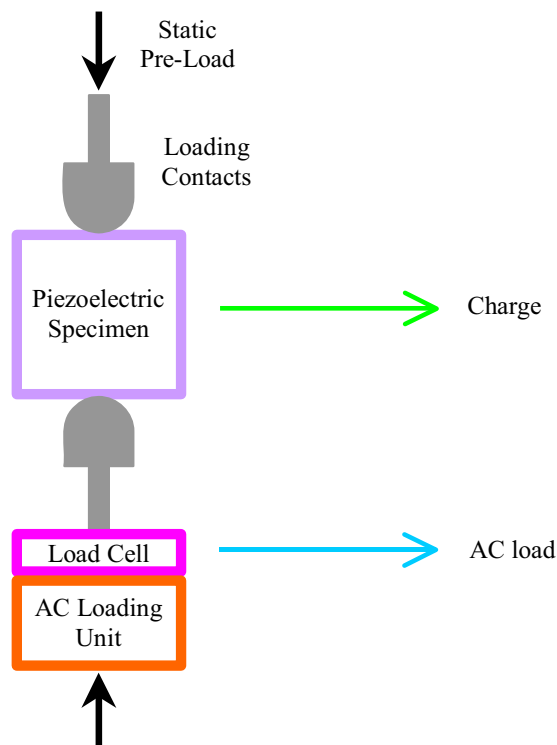
Figure 1 – Diagram showing the components of a simple Berlincourt system.

2 Objective

The objective of the work was to determine the current scatter in typical Berlincourt direct piezoelectric coefficient, d_{33}, measurements. An international inter-laboratory validation exercise was organised to obtain precision data for the Berlincourt method. 11 laboratories participated in the experiment performing 10 multiple measurements on each of 5 specimens of different materials and thicknesses. The participants, listed in Appendix A, were required to follow the instructions, as specified in Appendix B, which were supplied with the samples as closely as possible and report the results on the accompanying form, an example of which is shown in Appendix C.

3 Materials

Three materials, supplied by Morgan ElectroCeramics and Advanced Composites Ltd, were investigated at two different thicknesses, PC4D and ACL4040 being examples of "hard" materials with low d_{33} and PC5H being a "soft" material with high d_{33}:

- <u>material 1</u>: PC4D at 10mm thick,
- <u>material 2</u>: PC5H at 10mm thick,
- <u>material 3</u>: PC4D at 1mm thick,
- <u>material 4</u>: PC5H at 1mm thick,
- <u>material 5</u>: ACL4040 at 1mm thick.

All the ceramic specimens were of cylindrical geometry, nominally 10mm in diameter and were supplied to participants both poled and electroded, having been checked prior to dispatch for outliers. This was to ensure that outlying results could be traced back to the details of the measurements made by participants, rather than being caused by material or sample issues.

4   Piezoelectric Coefficient Results

The original raw data from all of the participants are presented in Table 1. The corresponding averages and standard deviations are shown in Tables 2 and 3.

Table 1 – Original data: d_{33} piezoelectric coefficient (pC/N)

Material / Laboratory	1			2			3			4			5		
1	282	282	282	563	565	585	274	266	265	535	452	470	239	229	230
	280	281	282	562	560	559	260	257	271	487	460	505	234	242	233
	279	283	280	562	563	563	262	258	258	460	461	461	239	237	237
		281			561			272			502			236	
2	295	295	291	584	588	585	322	323	330	576	569	583	279	278	275
	292	295	289	590	586	586	326	324	327	587	573	588	277	274	277
	294	291	293	586	594	593	325	325	328	578	581	578	278	274	278
		295			590			327			580			277	
3		150			553			185			328			166	
4	255.5		248.5	520.3		522.1	228.7		226.8	434.7		450.9	185.8		185.5
	265.7		273.7	515.6		503.3	215.5		216.3	446.1		410.7	193.7		182.9
	254.4		277.4	493.5		507.4	197.4		220.9	433.2		425.2	201.6		170.0
	258.0		262.4	515.5		523.5	230.7		212.8	407.7		426.1	189.6		189.0
	256.9		264.7	491.3		506.6	246.6		227.8	426.7		405.6	199.2		192.7
5	318	320	320	612	611	617	327	322	327	427	425	400	202	189	203
	317	319	321	606	616	608	325	320	321	437	404	410	211	216	188
	316	322	322	616	609	626	299	294	312	435	411	395	170	114	221
		318			628			280			383			219	
6	267	264	265	548	548	546	275	278	275	495	493	489	244	243	243
	258	264	266	547	545	545	271	267	270	505	529	514	246	248	243
7	671	700	699	1405	1431	1434	768	765	770	1430	1410	1353	645	652	657
	699	680	686	1427	1405	1409	792	764	755	1408	1363	1359	652	658	640
	681	690	704	1412	1440	1438	723	768	770	1351	1408	1350	657	640	653
		687			1440			752			1415			652	
8	286	286	286	588	595	586	305	303	308	558	539	558	263	266	261
	286	287	286	583	590	590	311	305	305	550	570	554	262	265	263
	286	283	285	586	591	587	306	305	302	551	549	537	259	265	261
		287			591			302			535			263	
9	289	290	289	581	580	577	317	316	317	463	454	494	268	270	267
	291	288	287	582	580	576	321	316	319	514	526	491	270	274	265
	291	289	291	581	582	581	320	318	317	443	475	486	264	263	269
		286			573			316			446			268	
10	289	287	289	564	564	570	310	315	315	440	489	460	268	258	263
	289	288	287	564	572	566	316	316	316	484	475	473	264	264	262
	287	288	288	565	569	571	313	315	314	479	477	485	256	265	261
		289			568			315			468			263	
11	288	289	281	585	585	583	323	319	325	524	500	488	274	276	275
	286	291	291	582	563	559	319	320	324	515	545	482	276	279	274
	286	293	290	576	571	567	321	323	325	509	511	513	275	275	275
		288			582			327			532			276	

Table 2 – Cell means: d_{33} piezoelectric coefficient (pC/N)

Material / Laboratory	1	2	3	4	5
1	281.2	564.3	264.3	479.3	235.7
2	293.0	588.2	325.7	579.3	276.7
3	150	553	185	328	166
4	261.72	509.91	222.35	426.69	189.00
5	319.3	614.9	312.7	412.7	193.3
6	264.0	546.5	272.7	505.6	244.5
7	689.7	1424.1	762.7	1384.7	650.6
8	285.8	588.7	305.2	550.1	262.8
9	289.1	579.3	317.7	479.2	267.8
10	288.1	567.3	314.5	473.0	262.4
11	288.3	575.3	322.6	511.9	275.5
Global mean	*326.8*	*659.3*	*343.3*	*580.6*	*286.3*

Table 3 – Cell standard deviations: d_{33} piezoelectric coefficient (pC/N)

Material / Laboratory	1	2	3	4	5
1	1.2	7.5	6.3	27.0	4.1
2	2.2	3.4	2.4	5.9	1.8
3	—	—	—	—	—
4	8.9	11.5	13.1	15.4	8.9
5	2.1	7.4	16.2	17.9	32.2
6	3.2	1.4	4.0	14.4	2.1
7	10.7	14.7	17.6	31.9	6.7
8	1.1	3.4	2.7	10.9	2.1
9	1.7	3.0	1.8	28.2	3.3
10	0.9	3.1	1.8	14.4	3.4
11	3.4	9.7	2.8	19.1	1.4

Participants followed the supplied round-robin recommendations only where adjustments were possible and current settings could be determined. In many cases, however, the targets could not be met due to limitations in the equipment control and so 'in-house' procedures were followed. The machine settings, often fixed by the manufacturer or difficult to control precisely by the user, were reported where these were known.

Cursory inspection of the data shows some clear irregularities:

- Laboratory 3 has provided only a single measurement for each material/specimen (laboratory variance cannot be calculated),
- Laboratory 7 is a marked outlier (all results are greater by approximately a factor of 2 times the global average).

Subsequent communications with participants uncovered that the test equipment at laboratory 7 had not been recalibrated after modifications were made to allow for the sample geometry. From the information provided by each participant on the detailed experimental set-up used, which is summarised and presented in Appendix D, it is apparent that laboratories 3 and 7 used AC load levels far outside the requested value and further that laboratory 3 used a far larger static pre-load than that recommended. This would take the material behaviour into the non-linear regime and so would lead to results which were not comparable.

Closer examination revealed that both these laboratories generally showed the greatest deviations from the global mean across all material types and thicknesses and were, unduly influencing the global mean, as shown numerically in Table 4 and graphically in Figure 2. This is evident by the dramatic change in global mean seen on excluding data from these two sites, and the increased randomness in the distribution of deviations from the revised global mean shown in Figure 2(b) compared to the original all inclusive data shown in Figure 2(a).

Table 4 – Cell deviation from original global means: d_{33} piezoelectric coefficient (pC/N)

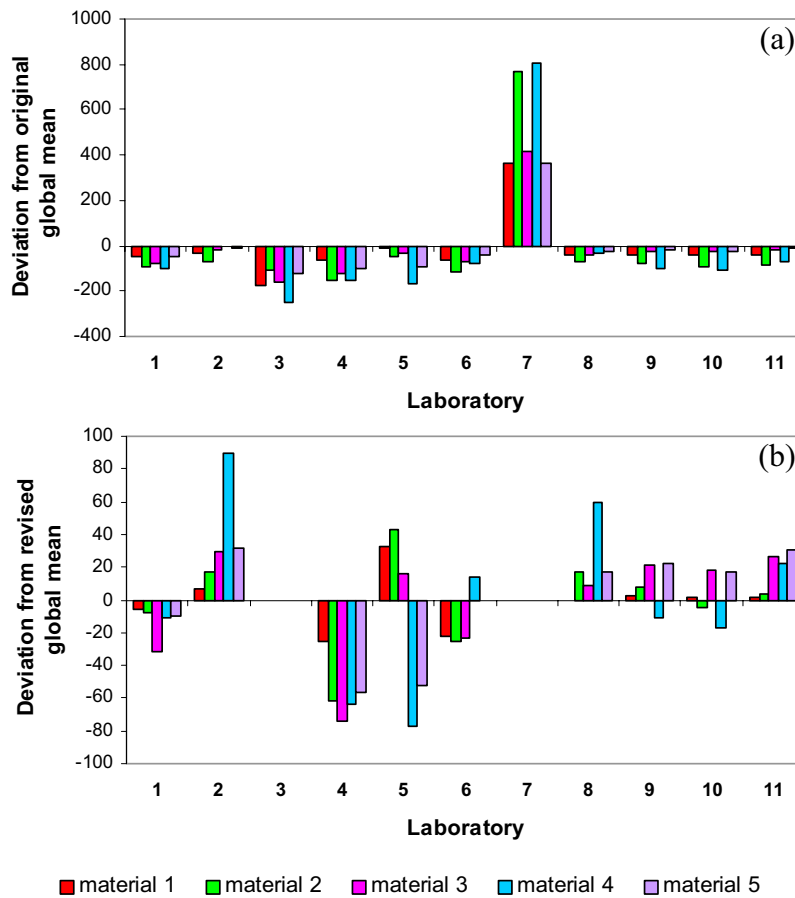| Material / Laboratory	1	2	3	4	5
1	-45.6	-95.0	-79.0	-101.3	-50.6
2	-33.8	-71.1	-17.6	-1.3	-9.6
3	-176.8	-106.3	-158.3	-252.6	-120.3
4	-65.08	-149.39	-120.95	-153.91	-97.3
5	-7.5	-44.4	-30.6	-167.9	-93.0
6	-62.8	-112.8	-70.6	-75.0	-41.8
7	362.9	764.8	419.4	804.1	364.3
8	-41.0	-70.6	-38.1	-30.5	-23.5
9	-37.7	-80.0	-25.6	-101.4	-18.5
10	-38.7	-92.0	-28.8	-107.6	-23.9
11	-38.5	-84.0	-20.7	-68.7	-10.8
Original global mean	*326.8*	*659.3*	*343.3*	*580.6*	*286.3*
Revised 9 site global mean	*286.6*	*571.6*	*296.4*	*490.1*	*245.3*

Figure 2 – Plot showing the deviations for individual participants of materials/thicknesses from (a) the respective original global means, and (b) the revised global means.

As a result of this combination of factors, data from both laboratories 3 and 7 were excluded from further analysis. This decision was further corroborated by the plot of the Mandel statistic based on comparison of the cell means presented in Appendix E.

The data from the remaining 9 participants was subsequently analysed according to ISO 5725-2 [2]. A typical example of the statistical output data from this analysis is shown in Appendix F.

5 Evaluation of Results

The retained data from the remaining 9 laboratories is represented in Figure 3 (a) – (e). These show several interesting features:

- Laboratories 4 and 5 generally exhibit larger experimental scatter than other participants,
- Material 4 (soft, thin sample) generally shows larger than average experimental scatter for all participants,
- Laboratory 1 has a potential individual outlier data point for material 2,
- Laboratory 5 has a potential individual outlier data point for material 5,
- Laboratory 4 consistently produces values markedly below the global average,
- Laboratory 2 consistently produces values markedly above the global average.
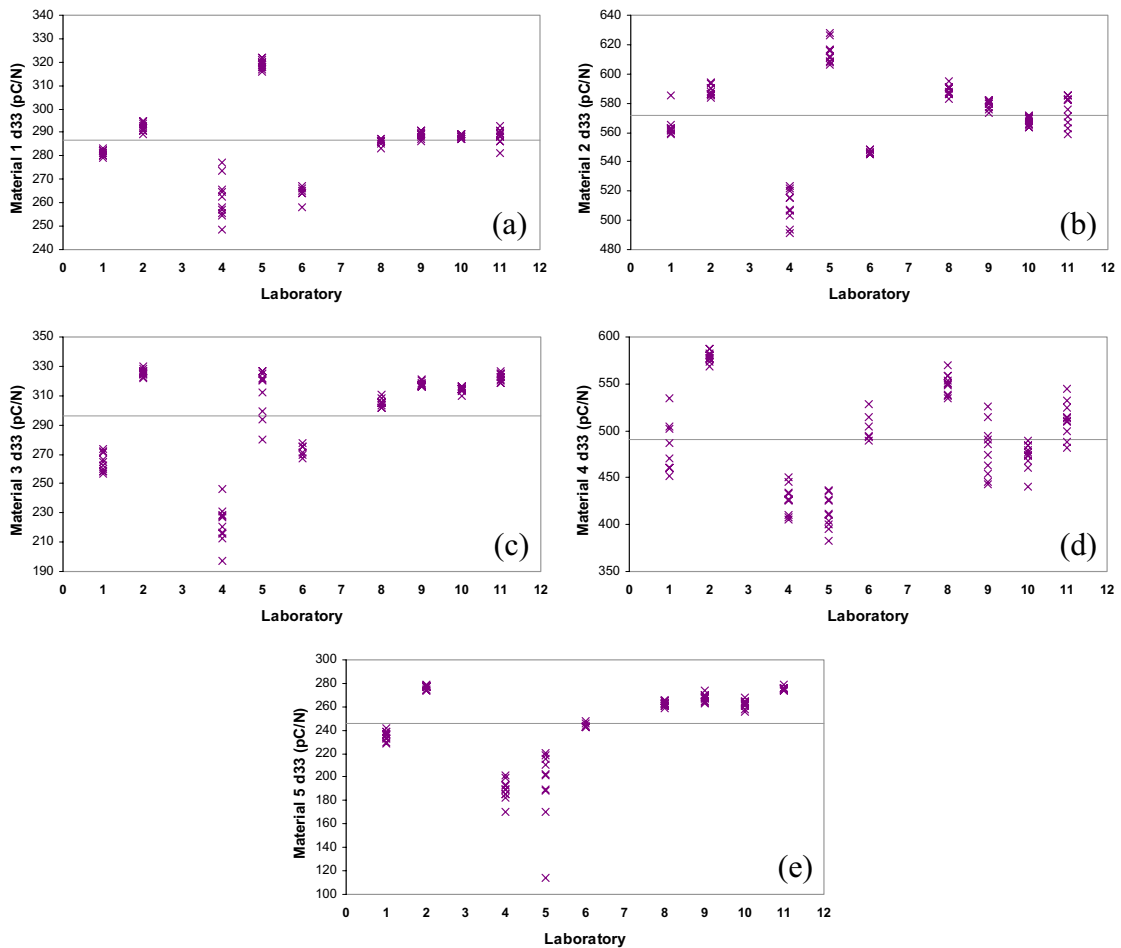
**Figure 3 – Individual measurement data points from retained participants:
(a) – (e) corresponding to materials 1 – 5 respectively,
horizontal lines represents the global mean.**

These features were further verified by Mandel's statistics performed on the data, used to assess laboratory consistency, charts of which are plotted in Figures 4 and 5. Mandel's 'h'-statistic is a measure of how the mean value for each laboratory and material differs from the average mean value amongst all participating laboratories and the 'k'-statistic is a measure of the standard deviation for each laboratory in relation to the sum of the variances of all the laboratories.
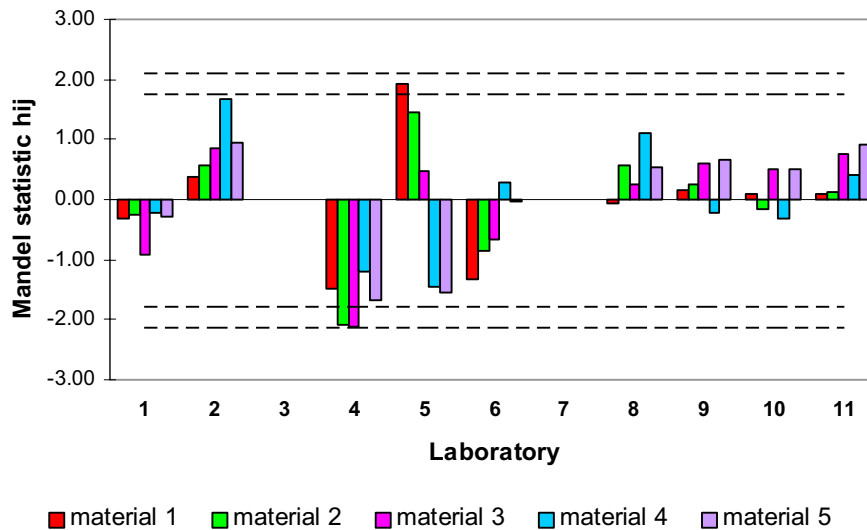

Figure 4 – Plot of Mandel's h-statistic for all included laboratories, dashed lines show the 1% and 5% significance levels.
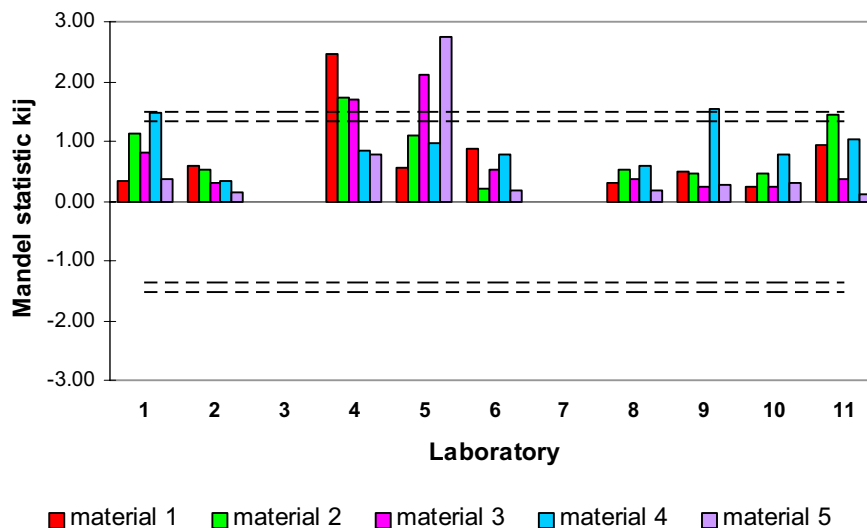

Figure 5 – Plot of Mandel's k-statistic for all included laboratories, dashed lines show the 1% and 5% significance levels.

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Tests on the cell <u>variances</u> for consistency and outliers (Cochran's - tests within laboratory variability by checking the homogeneity of variance across the laboratories) indicate the following outliers:

- Laboratory 4 - material 1,
- Laboratory 4 - material 2,
- Laboratory 5 and 4 - material 3,
- Laboratory 5 - material 5.

Tests on the cell <u>means</u> for consistency and outliers (Grubb's and Dixon's - test between laboratory variability based on the homogeneity of means across the laboratories) indicate no stragglers or outliers amongst any of the participants or materials.

The variance outliers highlighted above were retained for subsequent calculations. In this way, an estimate of the current levels of scatter could be determined for the measurements. By measuring the reproducibility/repeatability from different laboratories using different equipment and test procedures, the need for a standard procedure could be ascertained. In addition, there was no corroborative evidence in the result reports, from any of the participants, indicating that the outlying sets or data points differed from any of the other measurements made or were problematic in any way.

6   Precision Data

<u>Repeatability</u> refers to tests performed under conditions that are as constant as possible:

- during a short time interval
- in one laboratory
- by one operator
- using the same equipment.

<u>Reproducibility</u> refers to tests performed in widely varying conditions:

- in different laboratories
- with different operators
- using different equipment.

These values are therefore two extremes; repeatability measures the minimum, whilst reproducibility measures the maximum variability in results. Under totally controlled conditions (e.g. standardised procedure), repeatability and reproducibility can be defined as criteria by which to judge whether a difference between two test results can be ascribed to random fluctuation [3].

The general mean, *m*, the repeatability and reproducibility limits, *r* and *R*, and the repeatability and reproducibility standard deviations, s_r and s_R, are given in Table 5. Table 6 presents the same data as a percentage of the mean measured d_{33}.

Table 5 – Repeatability and reproducibility limits and standard deviations

Material	Repeatability Conditions		Reproducibility Conditions		Mean d_{33} (pC/N)
	s_r	r	s_R	R	m
1	3.63	10.15	16.85	47.19	286.62
2	6.64	18.60	30.45	85.26	571.61
3	7.74	21.66	36.08	101.02	296.35
4	18.42	51.59	56.99	159.58	490.36
5	11.67	32.68	36.03	100.88	245.33

Table 6 – Repeatability and reproducibility data expressed as a percentage of the mean

Material	Repeatability Conditions		Reproducibility Conditions	
	s_r (%)	r (%)	s_R (%)	R (%)
1	1.27	3.54	5.88	16.46
2	1.16	3.25	5.33	14.92
3	2.61	7.31	12.17	34.09
4	3.76	10.52	11.62	32.54
5	4.76	13.32	14.69	41.12

Repeatability or reproducibility standard deviations, s_r or s_R, are the standard deviation of test results obtained under repeatability or reproducibility conditions. These parameters measure the dispersion of the test results under repeatability conditions or the dispersion of the distribution of test results under reproducibility conditions.

The repeatability or reproducibility limits, r or R, are the values below which the absolute difference between two test results obtained under repeatability or reproducibility conditions can be expected to lie with 95% probability.

Thus the test method repeatability is very good, <5% for all materials, and there is no particular inherent problem with the test itself or any of the materials studied when a site/operator/equipment specific procedure is used. However, the reproducibility is poor, >5% for all materials and >10% for all thin samples (regardless of material type), suggesting a need to standardise the specifications to which the test is carried out across the different laboratories to ensure consistency and comparability of results. This fact is highlighted by the following analysis where a subgroup of the participant data was analysed separately.

7 Second Stage Analysis

Subsequent scrutiny of the procedures adopted by different participants, which is presented in Appendix D, revealed that a small group of four participants used conditions exactly, or very close to, those specified in the round-robin instructions.

This data subset was analysed to determine the effect of accurate test control on the repeatability and reproducibility of the test method and proposed procedure. This group comprised 4 participants: sites 2, 9 10 and 11. The results of the second stage evaluation are shown in Tables 7-9 and Figures 6 and 7.

Table 7 – Cell deviation from new global means: d_{33} piezoelectric coefficient (pC/N)

Material \ Laboratory	1	2	3	4	5
2	3.4	10.7	5.6	68.4	6.1
9	-0.5	1.8	-2.4	-31.7	-2.8
10	-1.5	-10.2	-5.6	-37.9	-8.2
11	-1.3	-2.2	2.6	1.0	4.9
9 site global mean	*285.6*	*570.5*	*295.3*	*490.7*	*245.3*
9 site global standard deviation	*16.0*	*28.9*	*34.3*	*54.6*	*34.3*
4 site global mean	*289.6*	*577.5*	*320.2*	*510.9*	*270.6*
4 site global standard deviation	*2.9*	*9.3*	*5.1*	*46.4*	*6.4*

Cochran's test on the cell <u>variances</u> indicates the following outliers and stragglers:

- Laboratory 11 - material 1 (outlier),
- Laboratory 11 - material 2 (outlier),
- Laboratory 9 - material 4 (straggler).

Grubb's and Dixon's tests on the cell <u>means</u> indicate no stragglers or outliers, as was found before on the larger data population.

The variance outliers highlighted above were retained for subsequent analysis as the Mandel's statistics plots, Figures 6 and 7, showed the variances in question were only just outside acceptable levels.
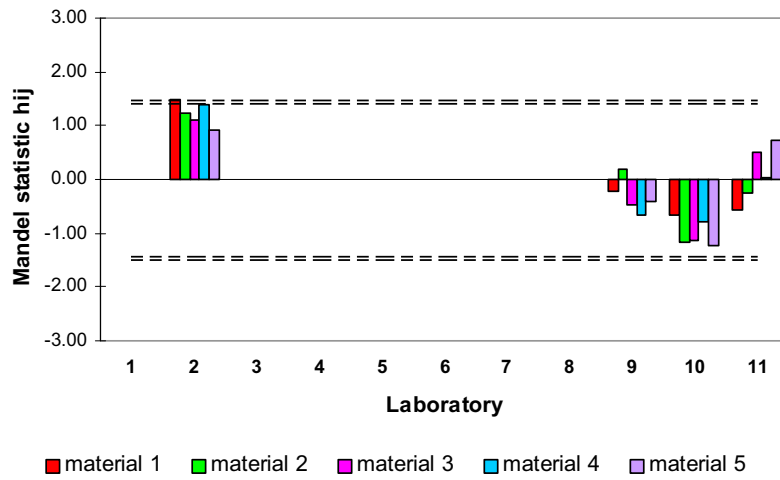
Figure 6 – Plot of Mandel's h-statistic for data subset, dashed lines show the 1% and 5% significance levels.
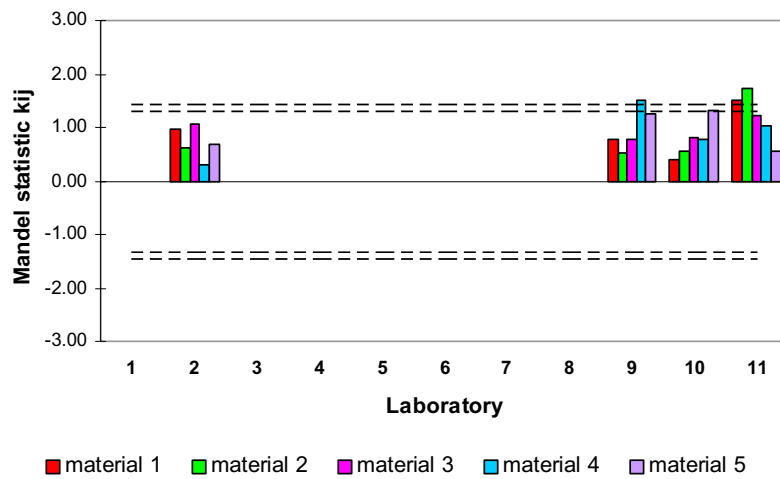

Figure 7 – Plot of Mandel's k-statistic for data subset, dashed lines show the 1% and 5% significance levels.

Table 8 – Repeatability and reproducibility limits and standard deviations

Material	Repeatability Conditions		Reproducibility Conditions		Mean d_{33} (pC/N)
	s_r	r	s_R	R	m
1	2.24	6.26	3.12	8.74	289.63
2	5.56	10.17	15.58	28.47	577.53
3	2.23	6.25	5.42	15.18	320.13
4	18.69	52.34	51.85	145.17	510.85
5	2.63	7.19	7.36	20.12	270.60

Table 9 – Repeatability and reproducibility data expressed as a percentage of the mean

Material	Repeatability Conditions		Reproducibility Conditions	
	s_r (%)	r (%)	s_R (%)	R (%)
1	0.77	2.16	1.08	3.02
2	0.96	1.76	2.70	4.93
3	0.70	1.95	1.69	4.74
4	3.66	10.25	10.15	28.42
5	0.97	2.66	2.72	7.44

This extended analysis, of the group of participants who used almost identical test set-up procedures, highlights several important points:

- Results are more tightly grouped around the global mean than in the larger data population and within site variances are more consistent,
- The reduction in the global standard deviations is greater for hard materials than soft, and is greater for thick samples than thin,
- Repeatability is improved to <1%,
- Reproducibility is greatly improved to <3%,
- Only marginal improvements were observed in repeatability and reproducibility for the thin sample of soft material (material 4).

This indicates that closely following a defined procedure greatly improves the reproducibility of results across a broad range of materials and sample geometries, operators and equipment manufacturer. Simultaneously, using the specified procedure also improved the repeatability of the measurements achieved.

The only exception is the particular problem posed by soft, thin materials in terms of both repeatability and reproducibility. This issue has been investigated previously where it was noted that the d_{33} of soft materials was very sensitive to the static pre-load applied and the d_{33} of thin materials was sensitive to thickness variations [1]. These effects are non-linear with stress and thickness and are caused by the high stresses and stress gradients in the region near

the contact points. These factors suggest that the utmost care must be taken when conducting measurements on soft material with thin sample geometries.

8   Concluding Remarks

The data presented in this report suggest the following:

- current practices of sites 2 and 7 were highly anomalous in comparison to the 'in-house' procedures of the remaining participants which was reflected in the results,

- current practice resulted in a laboratory bias being indicated for sites 2 and 4,

- current practices of sites 4 and 5 produced greater than average scatter for several of the materials studied,

- current practice shows good repeatability <5% for all materials and sample geometries,

- current practice shows adequate reproducibility ~5% for all bulk materials but poor reproducibility >10% for all thin samples regardless of material composition,

- adopting recommended practice produced a reduction in the global standard deviation of the data and the 'in-house' variances were more consistent,

- adopting recommended practice produced greater reductions in the global standard deviation for hard materials compared to soft, and for thick samples compared to thin,

- adopting recommended practice produced an improvement in repeatability to <1%,

- adopting recommended practice produced a significant improvement in reproducibility to <3% across almost the entire range of materials and geometries,

- adopting recommended practice produced only marginal improvements in repeatability and reproducibility for the soft, thin material samples; difficulties associated with the Berlincourt d_{33} measurements on thin, soft piezoelectric materials are well-known and documented (soft material response is non-linear with stress and, in the case of thin material, a proportionately greater volume is under high stress).

There is obviously scope for improving the direct method d_{33} measurement precision, using the Berlincourt technique, by the adoption of a standardised test procedure to control, record and report the values of parameters which significantly affect results. It has been shown in the data and analyses presented here that this would greatly improve the reproducibility of results across a broad range of materials and sample geometries, operators and equipment design.

The recommended procedure for direct piezoelectric coefficient measurement using the Berlincourt system requires close control of the factors listed, to the levels specified below:

- verify operation by **calibrating with a reference sample** e.g. quartz, LiNbO$_3$,

- conduct measurements at or close to **97 Hz** but not at multiples of mains frequency i.e. 50 Hz,

- use $\varnothing$ **2 mm hemispherical contacts**,

- **handle the sample with tweezers** where possible to prevent warming or allow the sample to equilibrate to room temperature after handling before making measurements,

- position the sample to give a positive d$_{33}$ reading,

- apply a pre-load of **10 N** $\pm$ 5% to the sample,

- apply an oscillating force of **0.1 N** RMS $\pm$ 10% to the sample.

- **note the temperature and humidity** of the testing environment as this affects the measured result.

9   References

1.   M. Stewart, W. Battrick and M. Cain, "Measuring Piezoelectric Coefficients using the Direct Method", NPL Measurement Good Practice Guide No. 44, 2001.

2.   BS ISO 5725 – 2:1994, "Accuracy (trueness and precision) of Measurement Methods and Results. Part 2. Basic Method for the Determination of Repeatability and Reproducibility of a Standard Measurement Method".

3.   BS ISO 5725 – 1:1994, "Accuracy (trueness and precision) of Measurement Methods and Results. Part 1. General Principles and Definitions".

APPENDIX A

VAMAS TWA24 Berlincourt Round-Robin Participants

The participants in the interlaboratory exercise were (alphabetically):

- Bath University, UK
- Cranfield University, UK
- EADS GmbH, Germany
- Instituto de Ciencia de Materiales de Madrid, Spain
- Laboratoire de Genie Electrique et Ferroelectricite, France
- Leeds University, UK
- National Physical Laboratory, UK
- Naval Undersea Warfare Center, USA
- Royal Military College of Canada, Canada
- Sensor Technology Ltd, Canada
- Toshiba Corp, Japan

APPENDIX B

Round-Robin Instructions Supplied to Participants

Instructions for d_{33} Round Robin Measurements with Berlincourt Type Meter

Objectives

The purpose of these tests is to examine some of the sources of scatter in typical Berlincourt type d_{33} measurements. There follows a few notes on how we carried out our measurements, so if you can closely match these it may eliminate some variability. Of course if your system cannot match these conditions, please use the conditions you would normally use and note this on the result sheet.

Experimental Procedure

- Verify the operation of your system as directed by your manufacturer, perhaps by measuring a reference sample provided with the system.

- We did our tests at 97Hz. Note the measurement frequency on the results sheet. If possible use 97Hz or close to this, but not a multiple of the mains supply frequency.

- Note the loading probe types used on the results sheet, in our comparison measurements we used hemispherical probes with a 2mm diameter.

- Note the room temperature for the measurements and if possible the humidity.

- Try not to handle the sample for too long, to prevent warming the sample.

- Position the sample to give positive reading, for our system the red plus mark facing down.

- If you know the pre-load applied to the sample, please give this. Our measurements used a pre load of 10N.

- If you know the AC measuring force of the system, please give this. Our Measurements used an AC force of around 0.1N rms.

- Make ten individual measurements on each of the five samples, or as many as you feel necessary.

- Report the values in pC/N.

Return the samples and results to NPL.

APPENDIX C

Form for Experimental Set-Up and Result Reporting Supplied to Participants

d$_{33}$ Round Robin Measurements Result Sheet

1. Information Requested

Your Organisations Name:	
Operators name:	
Date of Test:	
Manufacturers Name and Model Number:	
Frequency:	
Loading probe configuration:	
Room temperature:	
Humidity (if possible):	
Sample Pre Load (if known):	
AC measuring force (if known):	

2. Measurement results in pC/N

Sample #	Test 1	Test 2	Test 3	Test 4	Test 5

Sample #	Test 6	Test 7	Test 8	Test 9	Test 10

Note: The soft material is the light colour and the hard material a grey colour.

3. Return the samples and results to NPL

APPENDIX D

Information Provided by Participants on Detailed Experimental Set-Up

Site	Equipment Manufacturer	Frequency (Hz)	Contact Geometry	Ambient Temperature (°C)	Ambient Humidity (%)	Static Pre-Load (N)	AC Load (N - RMS)
1	Sensor Technology Ltd	100		23	43		
2	Channel Products Inc	95	5.6 mm ⌀ HS	22.5	49	10	0.1
3		97		22	55.5	1000	400
4	"In-house design"					14	1.5
5	Sensor Technology Ltd	97	1.25 mm ⌀ HS	21.2			0.1
6	Institute of Acoustics	110	HS	26.5			0.25
7		100	2 mm ⌀ HS	21			525
8	Channel Products Inc	100	5.5 mm ⌀ HS	23	47		
9	Take Control	97	4 mm ⌀ HS	22	40	10	0.1
10	Take Control	97	2 mm ⌀ HS	21		10	0.1
11	Take Control	97	2 mm ⌀ HS	24.2	25.1	10	0.1

HS – indicates hemi-spherical contacts

Shaded cells indicate missing information, either unknown or not supplied by participants.

APPENDIX E

Mandel's 'h'-Statistic for All Data from All Participants
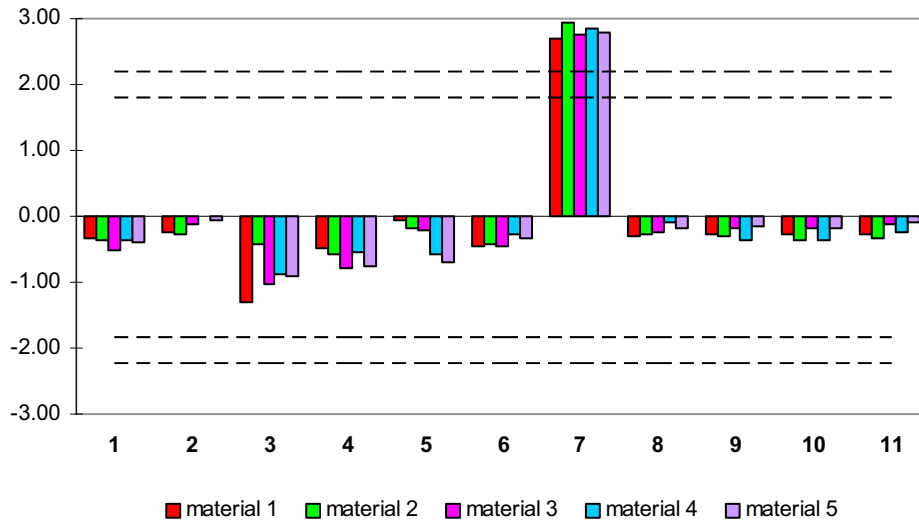
Measures how the mean value for each laboratory and material differs from the average mean value amongst all participating laboratories and shows that laboratories 7 and 3 (taking into account the lack of variance data for laboratory 3) are justifiably removed from the final analysis of the test method; as the presence of very deviant outliers can distort the view of the distribution as a whole.

APPENDIX F

Example of Statistical Data Analysis (material 5)

ISO5725- Part 2: 1994 Accuracy (trueness and precision) of measurement methods and results

User's data: MJLVAM5

laboratory i	lab 1	lab 2	lab 3	lab 4	lab 5	lab 6	lab 7	lab 8	lab 9	lab 10	lab 11
DATA	239.00	279.00		185.80	202.00	244.00		263.00	268.00	268.00	274.00
	229.00	278.00		185.50	189.00	243.00		266.00	270.00	258.00	276.00
maximum 25 labs (cols B to Z)	230.00	275.00		193.70	203.00	243.00		261.00	267.00	263.00	275.00
	234.00	277.00		182.90	211.00	246.00		262.00	270.00	264.00	276.00
maximum 10 replicates/lab (rows 10 to 19)	242.00	274.00		201.60	216.00	248.00		265.00	274.00	264.00	279.00
	233.00	277.00		170.00	188.00	243.00		263.00	265.00	262.00	274.00
ie. data goes into block B10..Z19	239.00	278.00		189.60	170.00			259.00	264.00	256.00	275.00
	237.00	274.00		189.00	114.00			265.00	263.00	265.00	275.00
	237.00	278.00		199.20	221.00			261.00	269.00	261.00	275.00
	226.00	277.00		192.70	219.00			263.00	260.00	263.00	276.00

no. labs. at current level pj	9.00										
no. replicates in cell nij	10.00	10.00		10.00	10.00	6.00		10.00	10.00	10.00	10.00
total number of data	86.00										
cell (lab) mean ybij	235.60	276.70		189.00	193.30	244.50		262.80	267.80	262.40	275.50
cell standard deviation sij	4.12	1.77		8.94	32.15	2.07		2.15	3.26	3.44	1.43
cell variance sij^2	16.93	3.12		79.96	1033.79	4.30		4.62	10.62	11.82	2.06
approx. lower 95% C.I. limit of mean	232.61	275.41		182.50	169.91	242.55		261.24	265.43	259.90	274.46
approx. upper 95% C.I. limit of mean	238.59	277.99		195.50	216.69	246.45		264.36	270.17	264.90	276.54

COCHRAN test on cell variances (see para. 7.3.3.2)											
max cell variance	1033.79										
sum of cell variances	1167.23										
Cochran critical 5% value		0.27									
Cochrans criterion	0.89		** outlier								
Cochran critical 1% value		0.31									

GRUBB test on cell means (see para. 7.3.4.)											
One outlying observation											
Grubb upper 5% critical value		2.22									
Grubb's statistic Gp(single high)	0.94										
Grubb's statistic G1(single low)	1.68										
Grubb upper 1% critical value		2.39									
Two outlying observations											
Grubb lower 5% critical value		0.15									
Grubb's statistic G (double high)	0.73										
Grubb's statistic G (double low)	0.16										
Grubb lower 1% critical value		0.09									

within-laboratory variance sr^2	136.21										
between-laboratory variance sL^2	1161.80										
reproducibility variance sR^2	1298.01										
repeatability standard deviation sr	11.67										
reproducibility standard deviation sR	36.03										
general mean mj	245.33										
repeatability limit r at 95% (2.8* sr)	32.68										
reproducibility limit R at 95% (2.8* sR)	100.88										

MANDEL statistics											
between-laboratory consistency hij	-0.29	0.94		-1.68	-1.55	-0.02		0.52	0.67	0.51	0.90
within-laboratory consistency kij	0.35	0.15		0.77	2.75	0.18		0.18	0.28	0.29	0.12

DIXON's test on cell means											
(see ISO5725 1987 para. 13)											
Q10	0.05										
Q11	0.05										
means with two largest set = null	235.60			189.00	193.30	244.50		262.80	267.80	262.40	
means with nulls + two smallest set = max	235.60	276.70	276.70	276.70	276.70	244.50	276.70	262.80	267.80	262.40	275.50
min-but-two mean	235.60										
max-but-two mean	267.80										
Q22	0.59										
Dixon 5% critical value		0.56									
Dixon statistic	0.05										
Dixon 1% critical value		0.67									