

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.

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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.

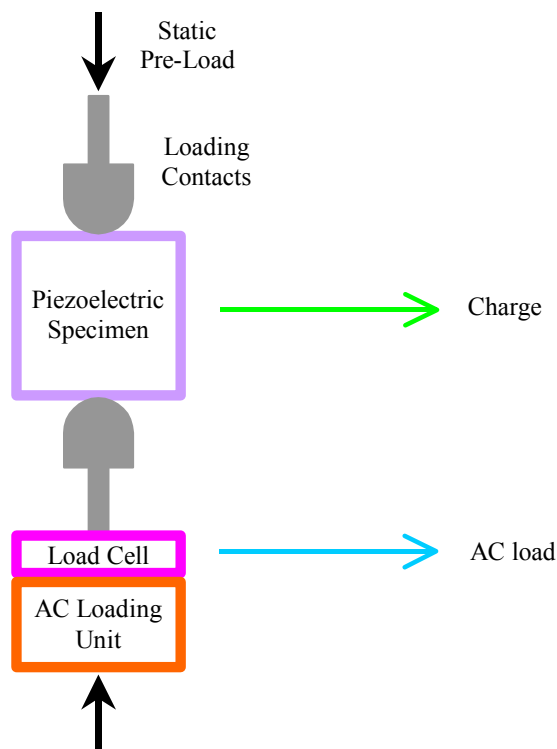


Figure 1 – Diagram showing the components of a simple Berlincourt system.

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

3 materials, supplied by Morgan ElectroCeramics and Advanced Composites Ltd, were investigated at 2 different thicknesses, PC4D and ACL4040 being examples of “hard” materials with low d_{33} and PC5H being a “soft” material with high d_{33} :

- material 1: PC4D at 10mm thick,
- material 2: PC5H at 10mm thick,
- material 3: PC4D at 1mm thick,
- material 4: PC5H at 1mm thick,
- material 5: 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	282 282 282 280 281 282 279 283 280 281	563 565 585 562 560 559 562 563 563 561	274 266 265 260 257 271 262 258 258 272	535 452 470 487 460 505 460 461 461 502	239 229 230 234 242 233 239 237 237 236
2	295 295 291 292 295 289 294 291 293 295	584 588 585 590 586 586 586 594 593 590	322 323 330 326 324 327 325 325 328 327	576 569 583 587 573 588 578 581 578 580	279 278 275 277 274 277 278 274 278 277
3	150	553	185	328	166
4	255.5 248.5 265.7 273.7 254.4 277.4 258.0 262.4 256.9 264.7	520.3 522.1 515.6 503.3 493.5 507.4 515.5 523.5 491.3 506.6	228.7 226.8 215.5 216.3 197.4 220.9 230.7 212.8 246.6 227.8	434.7 450.9 446.1 410.7 433.2 425.2 407.7 426.1 426.7 405.6	185.8 185.5 193.7 182.9 201.6 170.0 189.6 189.0 199.2 192.7
5	318 320 320 317 319 321 316 322 322 318	612 611 617 606 616 608 616 609 626 628	327 322 327 325 320 321 299 294 312 280	427 425 400 437 404 410 435 411 395 383	202 189 203 211 216 188 170 114 221 219
6	267 264 265 258 264 266	548 548 546 547 545 545	275 278 275 271 267 270	495 493 489 505 529 514	244 243 243 246 248 243
7	671 700 699 699 680 686 681 690 704 687	1405 1431 1434 1427 1405 1409 1412 1440 1438 1440	768 765 770 792 764 755 723 768 770 752	1430 1410 1353 1408 1363 1359 1351 1408 1350 1415	645 652 657 652 658 640 657 640 653 652
8	286 286 286 286 287 286 286 283 285 287	588 595 586 583 590 590 586 591 587 591	305 303 308 311 305 305 306 305 302 302	558 539 558 550 570 554 551 549 537 535	263 266 261 262 265 263 259 265 261 263
9	289 290 289 291 288 287 291 289 291 286	581 580 577 582 580 576 581 582 581 573	317 316 317 321 316 319 320 318 317 316	463 454 494 514 526 491 443 475 486 446	268 270 267 270 274 265 264 263 269 268
10	289 287 289 289 288 287 287 288 288 289	564 564 570 564 572 566 565 569 571 568	310 315 315 316 316 316 313 315 314 315	440 489 460 484 475 473 479 477 485 468	268 258 263 264 264 262 256 265 261 263
11	288 289 281 286 291 291 286 293 290 288	585 585 583 582 563 559 576 571 567 582	323 319 325 319 320 324 321 323 325 327	524 500 488 515 545 482 509 511 513 532	274 276 275 276 279 274 275 275 275 276

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	<i>326.8</i>	<i>659.3</i>	<i>343.3</i>	<i>580.6</i>	<i>286.3</i>

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

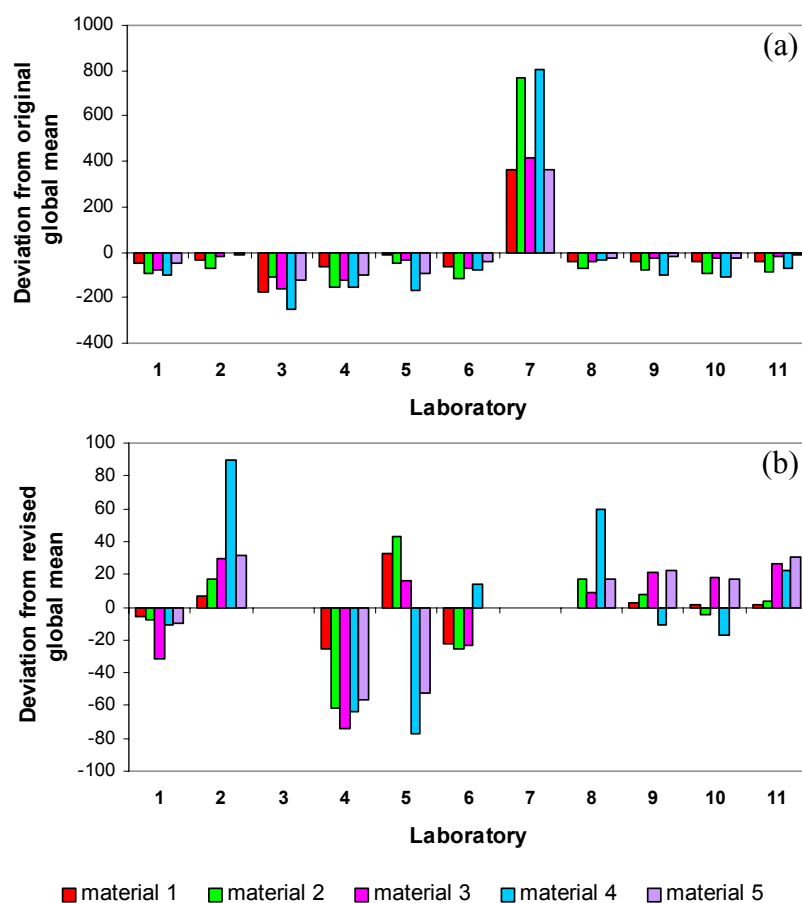


Figure 2 – Plot showing the deviations for individual participants of materials/thicknesses from (a) the respective original global means, and (b) the revised global means.

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.

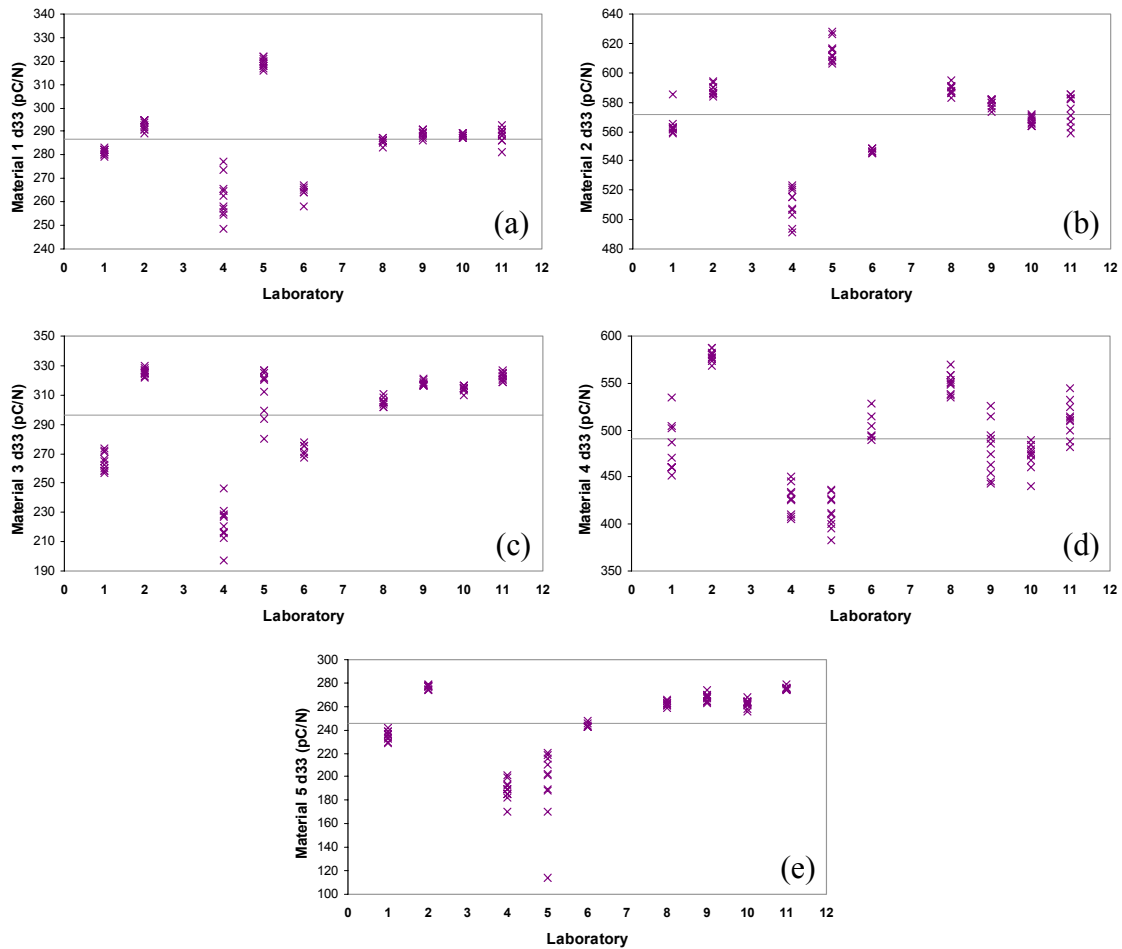


**Figure 3 – Individual measurement data points from retained participants:
(a) – (e) corresponding to materials 1 – 5 respectively,
horizontal lines represents the global mean.**

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.

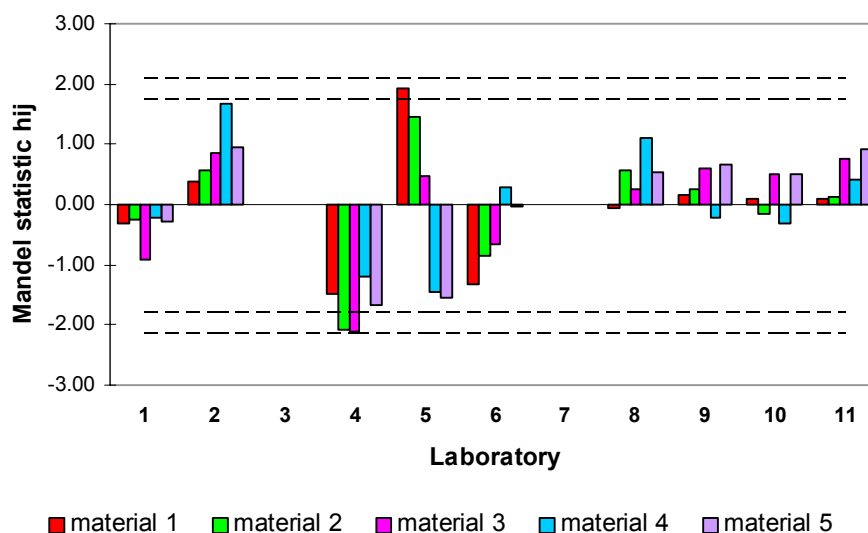


Figure 4 – Plot of Mandel's h-statistic for all included laboratories, dashed lines show the 1% and 5% significance levels.

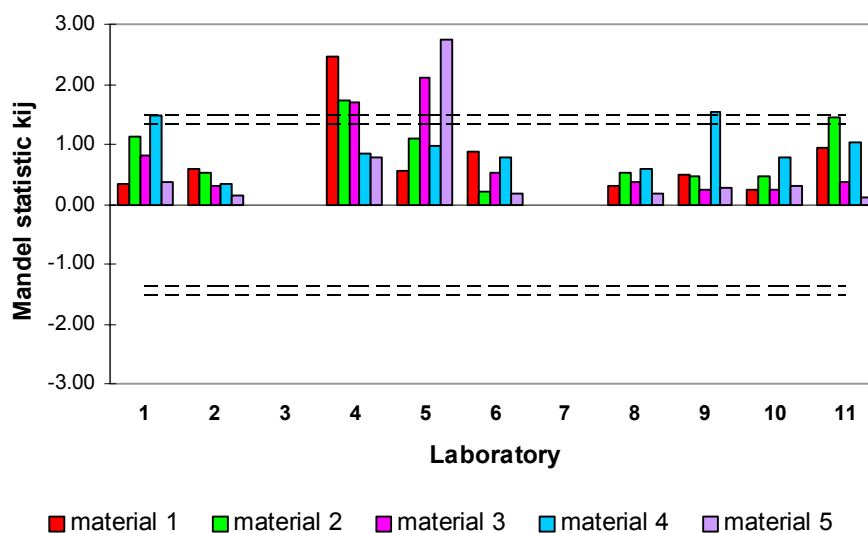


Figure 5 – Plot of Mandel's k-statistic for all included laboratories, dashed lines show the 1% and 5% significance levels.

Tests on the cell variances 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 means 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

Repeatability 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.

Reproducibility 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) m
	s_r	r	s_R	R	
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 variances 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 means 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.

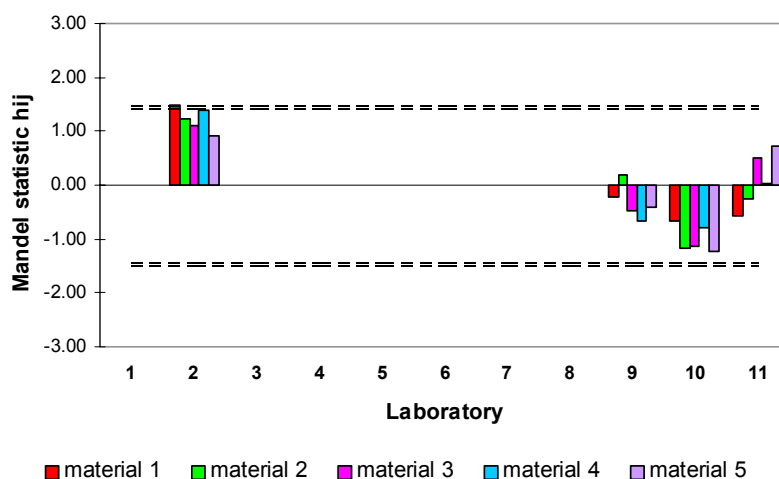


Figure 6 – Plot of Mandel's h-statistic for data subset, dashed lines show the 1% and 5% significance levels.

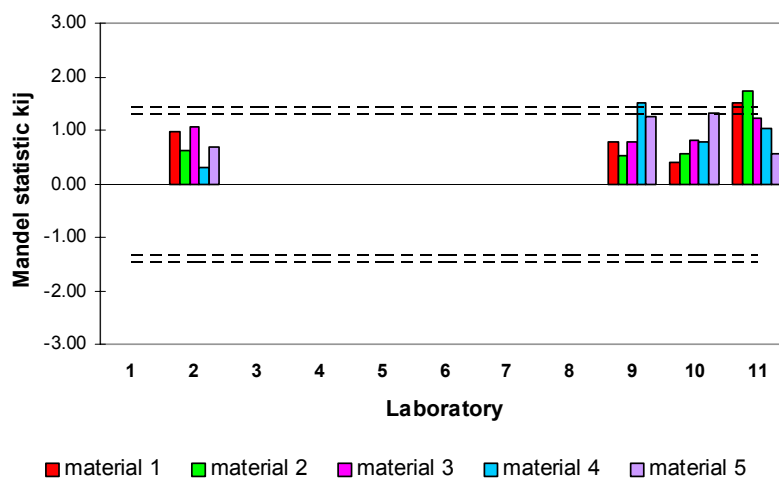


Figure 7 – Plot of Mandel's k-statistic for data subset, dashed lines show the 1% and 5% significance levels.

Table 8 – Repeatability and reproducibility limits and standard deviations

Material	Repeatability Conditions		Reproducibility Conditions		Mean d_{33} (pC/N) m
	s_r	r	s_R	R	
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 **Ø 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

d₃₃ 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

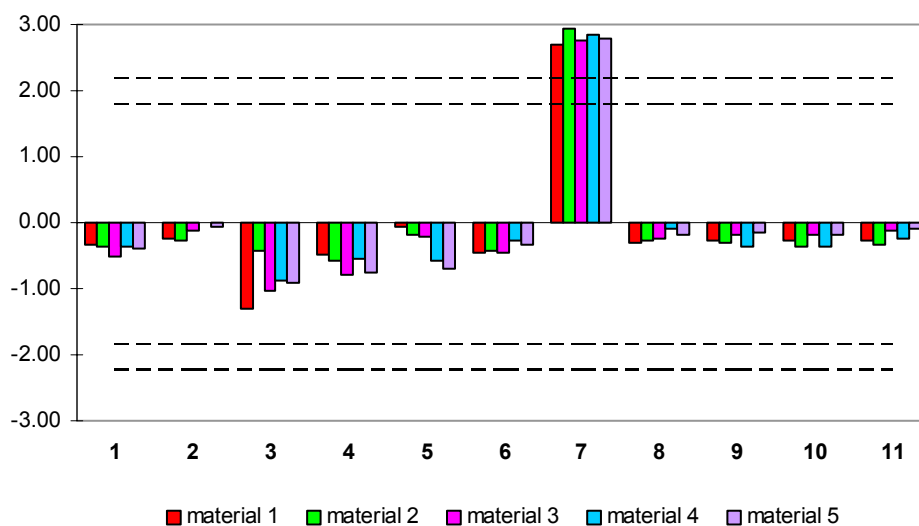
Site	Equipment Manufacturer	Frequency (Hz)	Contact Geometry	Ambient Temperature (°C)	Ambient Humidity (%)	Static Pre-Load (N)	AC Load (N - RMS)
1	Sensor Technology Ltd	100		23	43		
2	Channel Products Inc	95	5.6 mm Ø HS	22.5	49	10	0.1
3		97		22	55.5	1000	400
4	“In-house design”					14	1.5
5	Sensor Technology Ltd	97	1.25 mm Ø HS	21.2			0.1
6	Institute of Acoustics	110	HS	26.5			0.25
7		100	2 mm Ø HS	21			525
8	Channel Products Inc	100	5.5 mm Ø HS	23	47		
9	Take Control	97	4 mm Ø HS	22	40	10	0.1
10	Take Control	97	2 mm Ø HS	21		10	0.1
11	Take Control	97	2 mm Ø 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: M.J.L.VAM5											
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	229.00	279.00	185.00	202.00	244.00	262.00	268.00	268.00	268.00	274.00	
maximum 25 labs (cols B to Z)	229.00	278.00	185.00	189.00	243.00	266.00	270.00	268.00	276.00		
maximum 10 replicates/lab (rows 10 to 19)	230.00	275.00	192.70	202.00	242.00	261.00	267.00	267.00	275.00		
ie. data goes into block B10..Z19	234.00	277.00	182.90	211.00	246.00	262.00	270.00	264.00	276.00		
	242.00	274.00	201.60	216.00	248.00	265.00	274.00	264.00	279.00		
	233.00	277.00	170.00	180.00	243.00	263.00	265.00	262.00	274.00		
	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		
	236.00	277.00	192.70	219.00		262.00	268.00	262.00	276.00		
no. labs. at current level pj	9.00										
no. replicates in cell nj	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	6.94	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									