

The Analysis of Interlaboratory Testing Results of Rock Wool Products

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Received 03 September 2009; accepted 15 October 2009

The data obtained in interlaboratory comparison testing programme EA ILC MT2 devoted for testing compression behaviour of mineral wool slabs is discussed in this article. Fifteen laboratories from 11 European countries took part in this PT programme. The satisfactory fulfilling of tests by all laboratories excluding two was confirmed by statistical evaluation of test results. It is revealed the main causes of the test results scattering as well as the expedience to reconsider the Standard, which specifies the procedures for determining compression behaviour of thermal insulating materials.

Keywords: interlaboratory comparison testing, mineral wool slabs, compression behaviour, scattering of results.

1. INTRODUCTION

Compression behaviour is one of the most important properties of thermal insulating materials and products [1–4]. It is necessary to be certain in reliability of analysed data when comparing the results of different investigators. The most considerable complication came up when testing thermal insulating materials were made on the basis of mineral wool due their anisotropic (often crimped) structure [1]. Such structure complicates comparison of any test results [5]. Therefore the interlaboratory comparison testing in this field of investigations is very desirable. Statistical methods used for evaluation of given test results reliability [6–8] could allow to reveal the acceptable level of carrying out investigated experiments. However, such comparison data in scientific literature is scarce.

The purpose of this research was to assess the possible scattering of test results between different laboratories when they testing specimens cut out from the same mineral wool product and to estimate the most significant causes of this scattering. The results of proficiency testing programme EA ILC MT2 were used for this intention.

2. EXPERIMENTAL

The proficiency testing programme EA ILC MT2 “Testing of thermal insulating materials and products” devoted for testing compression behaviour of mineral wool slabs proposed by Lithuanian National Accreditation Bureau has been approved by the Laboratory Committee of European co-operation for Accreditation. Institute of Thermal Insulation of Vilnius Gediminas Technical University realized all technical aspects of this programme including the sample and specimen preparations, homogeneity testing, statistical evaluation of submitted results of participating laboratories and preparation of report [9]. All testing and evaluation of the results were finished in 2004.

Fifteen laboratories from Belgium, Denmark, Finland, Germany, Poland, Portugal, Slovakia, Slovenia and Spain including 3 Lithuanian laboratories took part in these

testings. All laboratories were supplied with five specimens of rock wool slabs and were asked to determine:

- compressive stress at 10 % relative deformation and compression modulus of elasticity according to EN 826 [10];
- specimen apparent density according to EN 1602 [11];
- organic content according to EN 13820 [12];
- expanded uncertainty of measurement of compressive stress at 10 % deformation and compression modulus of elasticity according to EA 4/02 [13].

Main characteristics for evaluation of test results were compressive stress $\sigma_{10\%}$ and compression modulus of elasticity E . Determination of density and organic content was necessary for additional control of homogeneity of specimens tested by laboratories.

The homogeneity of test pieces was investigated as well. The 160 test pieces of rigid mineral wool slabs were selected from 1340 ones in order to have a totality with mean value of apparent density fluctuating no more than $\pm 3 \text{ kg/m}^3$. These test pieces were divided into the five groups at most identical according to fluctuation of their density and organic content. About 40 % of test pieces from each group were selected into first set intended for homogeneity testing. The rest set was intended for testing by laboratories.

It was ascertained that repeatability standard deviation S_r and reproducibility standard deviation S_R [8] of density in these sets and between sets are like and both coefficients of variation are about 1.1 %, i. e. both sets of test pieces are almost identical according to their densities.

Test pieces from first set were squared to $200 \text{ mm} \times 200 \text{ mm}$ and prepared for detailed testing in providing laboratory. Nominal thickness of the test pieces was 80 mm.

The compression tests were carried out according to [10] on computerized testing machine H10KS (Hounsfield, UK) with force measurement error of 1 N–11 N. The accuracy of deformation measurements was 0.01 mm.

The results of homogeneity control by density, organic content, compressive stress and compression modulus of elasticity are presented in Table 1.

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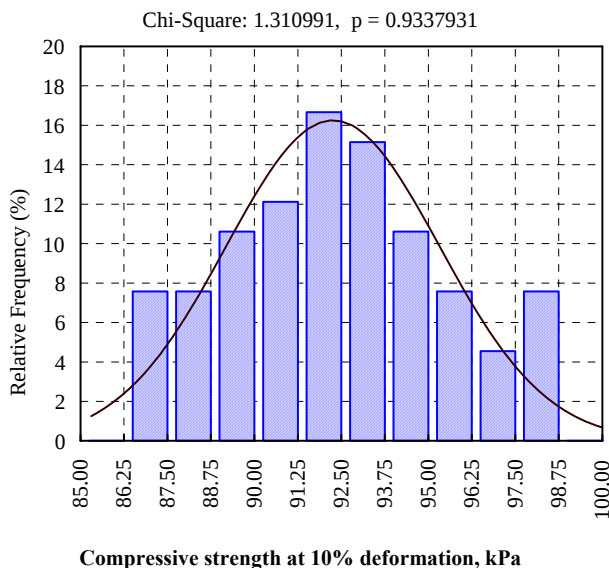
Table. 1. Results of the homogeneity testing

Group No	Density, kg/m ³		Organic content, % mass		Compressive stress at 10 % deformation $\sigma_{10\%}$, kPa		Compression modulus of elasticity E , kPa	
	mean value	standard deviation	mean value	standard deviation	mean value	standard deviation	mean value	standard deviation
1	154.5	3.08	3.50	0.30	94.0	3.48	1760	102.5
2	154.8	1.91	3.39	0.17	92.2	4.13	1737	94.5
3	154.8	2.34	3.43	0.17	92.7	2.85	1760	74.8
4	154.9	2.59	3.56	0.29	91.1	2.19	1645	102.7
5	154.8	3.35	3.46	0.19	91.6	2.57	1636	93.3
Total mean value	154.7	–	3.48	–	92.3	–	1699	–
Repeatability standard deviation S_r	2.83 (1.83 %)		0.24 (6.8 %)		2.98 (3.2 %)		94.6 (5.6 %)	
Reproducibility standard deviation S_R	2.72 (1.76 %)		0.24 (6.8 %)		3.10 (3.4 %)		111.7 (6.6 %)	

It is possible to state that homogeneity by density is good by organic content is satisfactory taking into account a nature of mineral wool products.

The dependences between density and $\sigma_{10\%}$ as well as between density and E were checked by regression analysis [14] of all initial data. It was determined that there is no reliable correlation in the investigated density interval.

Fig. 1 presents histograms of the observed frequency distribution of $\sigma_{10\%}$. The hypothesis about the normality of test results presented in Table 1 was checked and confirmed using χ^2 criterion [15].

**Fig. 1.** Histograms of the observed frequency distribution of $\sigma_{10\%}$ values obtained in the homogeneity testing

Homogeneity within and between groups of the test specimens was in addition investigated using the Mandel's h and k statistics [8]. Between groups consistency Mandel's statistic showed that the view of received $\sigma_{10\%}$ and E results is not unusual (received positive values are

approximately equal to negative ones). All h values are smaller corresponding Mandel's indicators at the 1 % and 5 % significance levels.

The standard deviations of group results were used for the calculation of Mandel's groups consistency statistic k . All five k values were smaller than critical value and showed a good stability of test results homogeneity.

On the basis of presented homogeneity tests, it was possible to state that the specimens of all 5 groups are comparatively homogeneous taking into account a nature of mineral wool products.

Therefore although standard deviations of compressive stress and compression modulus of elasticity are relatively considerable, it was decided to distribute test pieces to participating laboratories and to take into account this fact while evaluating test results. Participating laboratories were supplied by one test piece from each group.

3. TESTS RESULTS AND DISCUSSION

The averaged test results of participating laboratories are presented in Table 2. There is given the repeatability standard deviation S_r , which is the standard deviation of received results under repeatability conditions (same laboratory, same equipment and same staff) as well as the reproducibility standard deviation S_R , which is the standard deviation of received results under reproducibility conditions (different laboratories, different equipment and different staff) [8]. There are also presented in this Table the medians $med(x)$ of results, which were used in exchange for mean values, and corresponding values MAD , which were used in exchange of standard deviations [13]. MAD is the central tendency of absolute deviations ($x_i - med(x)$). The advantage of this order statistic-median is independence of statistical analysis of measurement results upon the given accidental extreme values. The results which were indicated as outliers by initial statistical evaluation were excluded when calculating $med(x)$ and MAD .

Statistical analysis for all results presented by laboratories included evaluation of:

Table 2. Results of the participating laboratories

Code of laboratory	Density, kg/m ³		Organic content, % mass		Compressive stress at 10 % deformation $\sigma_{10\%}$, kPa		Compression modulus of elasticity E , kPa	
	mean value	standard deviation	mean value	standard deviation	mean value	standard deviation	mean value	standard deviation
01	152	1.3	3.42	0.23	72.4	2.5	787	102
02	154	3.4	–	–	82.3	4.7	1583	132
04	150	2.1	–	–	89.9	2.6	1335	171
05	156	3.2	3.37	0.35	86.3	4.4	1658	153
06	155	2.4	3.64	0.09	84.1	5.3	–	–
07	154	3.4	–	–	87.9	5.1	1629	201
08	157	1.14	3.61	0.24	91.5	4.25	–	–
09	155	0.9	3.51	0.07	93.1	5.0	1762	144
10	157	2.0	–	–	92.1	4.2	2010	178
11	153	2.7	3.67	0.23	88.2	4.7	1648	85
13	152	2.0	–	–	83.6	1.5	–	–
14	154	3.1	–	–	84.4	3.3	1505	33.9
15	152	2.2	3.78	0.075	81.6	4.3	1431	103
16	155	3.0	–	–	90.2	5.8	1603	234
17	155	3.4	–	–	88.8	1.5	–	–
Total mean value	154.1	–	3.57	–	87.1	–	1541	–
Repeatability standard deviation S_r	2.5 (1.6 %)		0.21 (5.8 %)		4.1 (4.7 %)		150 (9.7 %)	
Reproducibility standard deviation S_R	3.0 (1.9 %)		0.24 (6.6 %)		6.7 (7.7 %)		333 (21.6 %)	
$med(x)$	154.5		3.59		88		1584	
MAD	2.6 (1.7 %)		0.13 (3.6 %)		3.6 (4.1 %)		130 (8.2 %)	

– normal distribution hypothesis using software STATISTICS [15];

– laboratory's individual results suitability using Grubbs' test [8];

– hypothesis of within – laboratory precision of variances using Cochran' test [8];

– Mandel's between and within laboratory consistency statistics (h and k), grouped by laboratories [8];

– comparability of mean values of results using distribution of their variation limits evaluated according to Student [15];

– central tendency of presented testing results according to positional statistics $med(x)$ and central tendency of absolute deviations determined by MAD [16].

The statistically evaluated results of density determination are presented in Fig. 2, a. These results are close to the results of providing laboratory (see Tables 1 and 2). Therefore fluctuation of apparent density could not have a noticeable influence on the values of $\sigma_{10\%}$ and E determined by the laboratories. Detailed analysis of these density results according to full statistical analysis programme mentioned above testified that all results of all laboratories were satisfactory.

The averaged results of organic content determination are close to the values determined by PT provider (see Tables 1 and 2). A part of results presented by two

laboratories (see Fig. 2, b) are out of limit $\pm 3S_R$. However detailed statistical analysis confirmed its as suitable for further evaluations.

Results of participating laboratories usually are evaluating using Z – scores [7, 17]:

– $|Z| < 2$ the result is considered satisfactory;

– $2 \leq |Z| \leq 3$ the results is considered questionable;

– $|Z| > 3$ test result is considered unsatisfactory,

where $Z = \frac{\bar{x}_i - \tilde{x}}{S_R}$; $\bar{x}_i$ – is mean value of results presented

by laboratory; $\tilde{x}$ – is consensus (mean) value of results presented by all laboratories; S_R – reproducibility standard deviation.

In our case $Z = \frac{\bar{x}_i - med(x)}{MAD}$.

Calculated Z values for organic content are given in Fig. 3, a. They confirmed that results of all laboratories are satisfactory. However high value of S_R are indicating that it is not worth-while to evaluate individual values of this index because of the problems in the uniform distribution of organic binder in the tested products. Never the given results allow to state that noted fluctuation of organic content in the specimens could not have essential influence on the averaged results of compressive stress at 10 % deformation and compression modulus of elasticity.

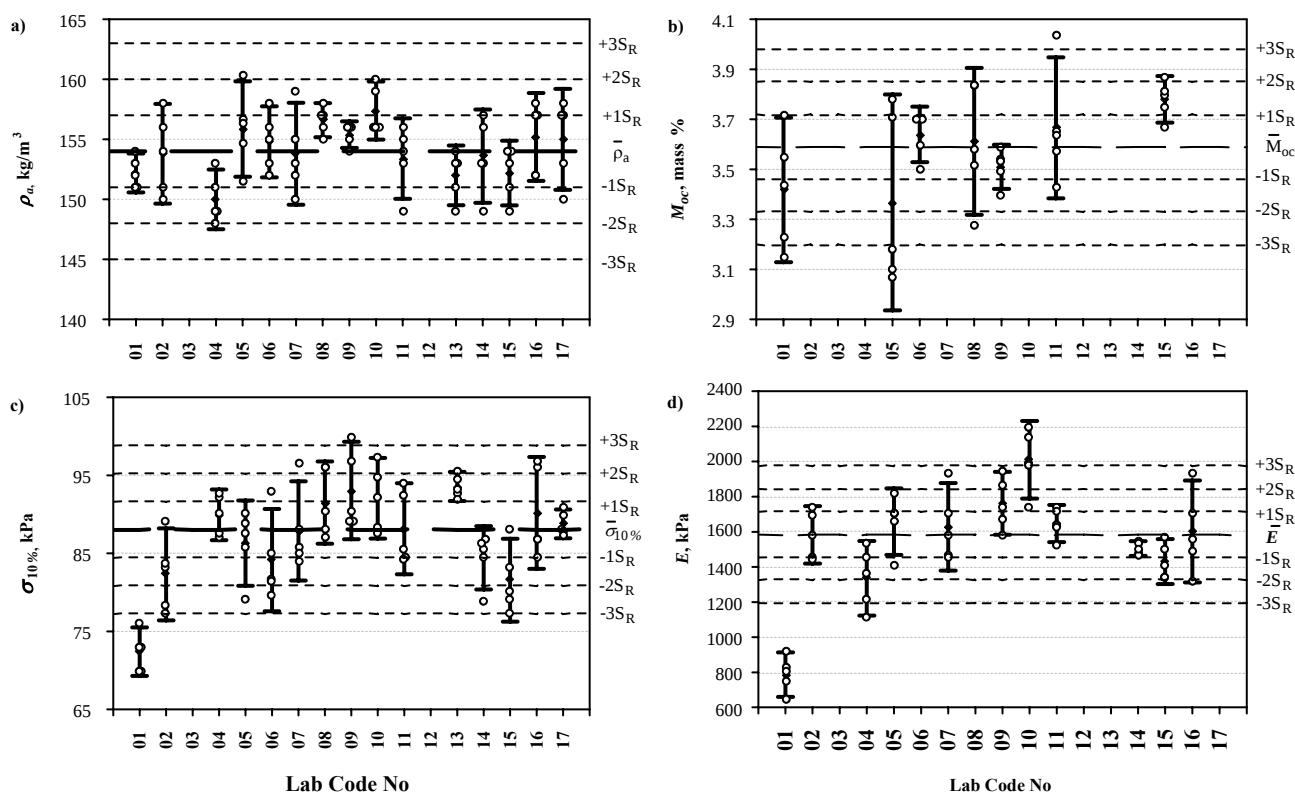


Fig. 2. Statistically evaluated results: a – apparent density; b – organic content; c – compressive stress at 10 % deformation; d – compression modulus of elasticity. The MAD are used in corresponding limits $\pm 1S_R$, $\pm 2S_R$ and $\pm 3S_R$ instead of S_R .

The averaged and statistically evaluated results of compressive stress at 10 % deformation $\sigma_{10\%}$ are presented in Table 2 and Fig. 2, c. Detailed analysis of these results according to full statistical analysis programme revealed that results of laboratory No 01 are outliers and could not be used for calculation of consensus value. This result is not satisfactory and according to Z – score (see Fig. 3, b). Some individual results of other laboratories exceed the $\pm 2S_R$ limit but they are not considered to be questionable or unsatisfactory, as they can be conditioned by specimen homogeneity scattering. However it may be stated that direct regularities between fluctuations of density, organic content and $\sigma_{10\%}$ values (see Fig. 2, a, b and c) were not observed for results of these laboratories. It is obvious that these extreme values caused considerable value of *MAD* as compared with S_R (see Table 2). The difference between *med*(*x*) of $\sigma_{10\%}$ and corresponding mean value given by providing laboratory in homogeneity testing is equal 5.8 %.

The averaged and statistically evaluated results of compression modulus of elasticity are presented in Table 2 and Fig. 2, d. It can be stated that scattering of these results is greater than in case of $\sigma_{10\%}$. It is seen that *MAD*, which excludes influence of extreme values, is 2.6 times lower than corresponding S_R . Full statistical analysis revealed that results of laboratories No 01 and 10 are outliers. They were not used for further calculations including consensus value and *MAD*. These results are not satisfactory when calculating Z – score (see Fig. 3, c) too.

The difference between *med*(*x*) of *E* and corresponding mean value given by providing laboratory is equal 9.7 %. Some individual results of some other laboratories slightly exceed the $\pm 2S_R$ limit. As in case of $\sigma_{10\%}$ there are not

stated direct regularities between fluctuation of density, organic content, $\sigma_{10\%}$ and *E* values (see Fig. 2) for results of these two laboratories.

The laboratories were asked to present measurement uncertainties of results $\sigma_{10\%}$ and *E* according to EA 4/02 [13]. The received data is presented in Table 3.

The given expanded uncertainties of compressive stress measurement fluctuate from 0.36 kPa to 12.4 kPa and differ from each other by up to 34 times. The expanded uncertainties of compression modulus of elasticity measurement fluctuate too and differ from each other by up to 13 times.

Expanded uncertainties of $\sigma_{10\%}$ and *E* measurement in specimen homogeneity tests were estimated by PT provider equal respectively 0.8 kPa and 72 kPa.

Information received from filled questionnaires presented to providing laboratory by participating laboratories show, that most of the laboratories used testing machines, which are directly controlled by software and guarantee loading and displacement measurements with good accuracy respectively $\pm 0.5\%$ and ± 0.01 mm. A very significant scattering value of uncertainty of measurements is clearly caused by the peculiarities and inexactitudes of laboratory procedures for evaluation of these uncertainties. Therefore it would be expedient to present more precise information about the accuracy of measurement and their evaluation in the new EN 826 edition or to prepare harmonized instruction for evaluation of uncertainty of concrete measurements. It is a pity that a small number of participants made the statistical data evaluation difficult and did not allow to decrease slightly the corresponding S_R .

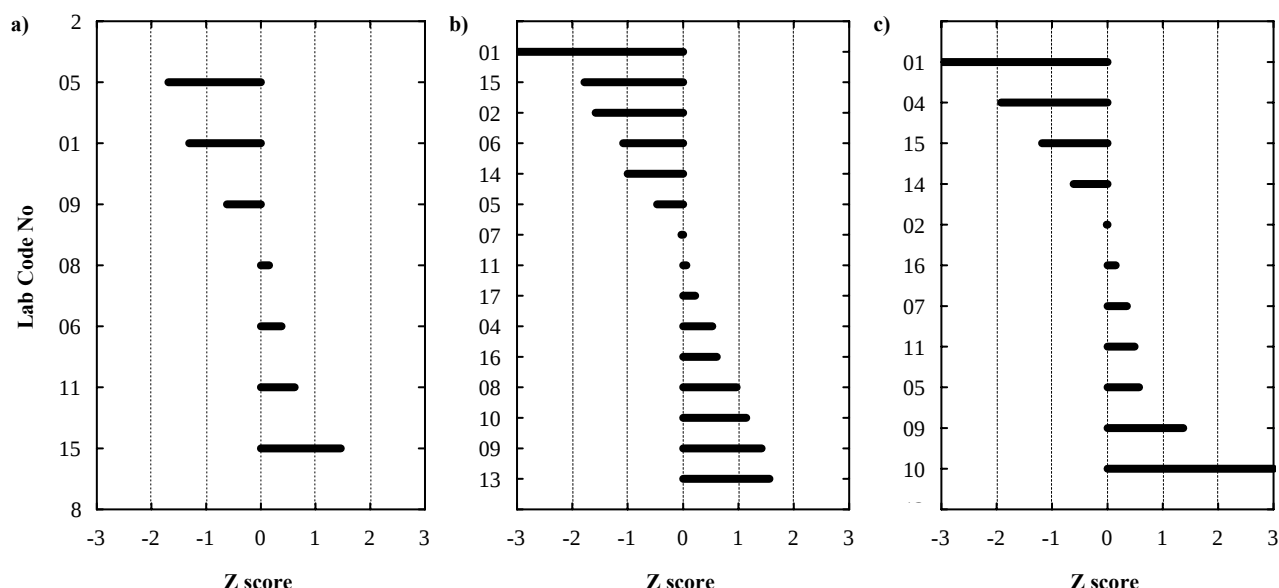


Fig. 3. Z scores for the mean values of: a – organic content; b – compressive stress $\sigma_{10\%}$; c – compression modulus of elasticity E

Table 3. Expanded measurement uncertainties presented by laboratories

Lab code	01	02	04	05	06	07	08	09	10	11	13	14	15	16	17
$U(\sigma_{10\%})$, kPa	0.36	1.3	–	4.6	10.0	–	12.4	10.54	–	2.34	1.46	7.0	0.4	3.4	1.0
$U(E)$, kPa	11.8	74	–	–	–	–	–	133.6	–	–	–	82	10	–	–

Table 4. Comparison of reproducibility participant results with those presented in EN 826:1996

Data	Parameter	Mean value, kPa	The reproducibility standard deviation S_R , kPa (%)	The 95 % reproducibility limit R , kPa (%)
Accuracy of measurements given in EN 826:1996	$\sigma_{10\%}$ E	95...230 2500...8500	$S_R = 3\%$ $S_R = 10\%$	9 % 25 %
EA ILC MT2, results of labs (see Table 2)	$\sigma_{10\%}$ E	$med(x) = 88$ $med(x) = 1584$	$MAD = 3.6$ kPa (4.1 %) $MAD = 130$ kPa (8.2 %)	10.1 kPa (11.5 %) 364 kPa (23.0 %)
EA ILC MT2, homogeneity testing (see Table 1)	$\sigma_{10\%}$ E	92.3 1699	$S_R = 3.1$ kPa (3.4 %) $S_R = 112$ kPa (6.6 %)	8.7 kPa (9.4 %) 313 kPa (18.4 %)

values, i.e. to narrow the intervals of satisfying test performance.

Table 4 presents the comparison of reproducibility of $\sigma_{10\%}$ and E results of the participating laboratories and the corresponding results of unidentified three object testing in ten laboratories indicated in EN 826 [10]. The corresponding information about MT2 specimen homogeneity test by providing laboratory is also presented in this table.

It can be seen that the characteristics of reproducibility of EA ILC MT2 participant results are close to those presented in EN 826.

It was shown above that fluctuation of specimen apparent density and organic content could not have important influence on specimen compression behaviour. The observed scattering of results apparently could be determined by uneven distribution of organic binder in rock wool specimens and more considerably by the crimping degree of rock wool layer before hardening, which may not easily quantitatively evaluated [18].

The additional scattering of compressive stress and compression modulus of elasticity between laboratories

could be influenced by the non-coordination between the possibilities of the used testing machines and requirement of EN 826:1996. This standard edition was more suitable to the machines of older generation, when the written force – displacement curves were used, and when the displacement segments X_c and X_{10} (see [10]) were evaluated graphically. From all participating laboratories of this program only 2 used the machines without software. Other labs used testing machines with software produced by 8 different companies. It is known that the software of different companies evaluated the initial points of displacement segments X_e and X_{10} a little bit differently, as well as the conventional elastic zone of force – displacement curve. This could influence differences of the results of $\sigma_{10\%}$ and especially E between the laboratories. Therefore the detailed revision of all basic sections, adapting them to modern testing equipment shall be welcomed in the new EN 826 edition. After this adapting all corresponding software for determination of $\sigma_{10\%}$ and E shall be corrected.

3. CONCLUSIONS

Almost all laboratories fulfilled tests of rock wool products satisfactory in the frame of setted requirements of experiments.

The accuracy of testing mineral wool products is restricted not only by essential inhomogeneity of these objects but largely by non-coordination between the possibilities of the used testing machines with different software and requirement of acting standard which specifies the equipment and procedures for testing.

The uncertainties of measurement of mineral wool product characteristics shall be evaluated only on the basis of harmonized instructions.

Acknowledgments

The authors acknowledge the contribution of participating laboratories, and Ms. Irena Vasaitiene (Lithuanian National Accreditation Bureau) who helped in the organization of EA ILC MT2 programme and its representation in EA Laboratory Committee.

REFERENCES

1. **Gnip, I. J., Vaitkus, S., Kersulis, V., Vėjelis, S.** Predicting the Deformability of Mineral Wool Slabs under Constant Compressive Stress *Construction Building Materials* 23 2009: pp. 1928–1934.
2. **Gnip, I., Kersulis, V., Vaitkus, S., Vėjelis, S.** Assessment of Strength under Compression of Expanded Polystyrene (EPS) Slabs *Materials Science (Medžiagotyra)* 10 (4) 2004: pp 326–329.
3. **Chistyakov, A. M.** Lightweight Multilayer Enclosing Structures. Moscow, Stroyizdat, 1987: 240 p. (in Russian).
4. **Vaitkus, S., Gnip, I., Kersulis, V., Vėjelis, S.** Prediction of Creep Strain of the Expanded Polystyrene (EPS) in Long-term Compression *Materials Science (Medžiagotyra)* 13 (4) 2007: pp. 293–298.
5. **Wiglus, R., Jarnuskiewicz, J., Sitko, E., Nikel, G.** Interlaboratory Comparison Experiment on the Determination of Formaldehyde Emitted from Mineral Wool Board Using Small Test Chambers *Building and Environment* 35 2000: pp. 53–57.
6. **Luping, T., Schouenborg, B.** Methodology of Inter-comparison Tests and Statistical analysis of Test Results. Nordtest Project No. 1483-99. Swedish National Testing and Research Institute, Boras, 2000: 40 p.
7. ISO 13528:2005 E. Statistical Methods for Use in Proficiency Testing by Interlaboratory Comparisons.
8. Final Report of EA ILC MT2. Testing of Thermal Insulating Materials and Products. Institute of Thermal Insulation of VGTU, Lithuanian National Accreditation Bureau, Vilnius, 2004: 95p.
9. EN 826:1996 E. Thermal Insulating Products for Building Applications. Determination of Compression Behaviour.
10. EN 1602:1996+AC:1997 E. Thermal Insulating Products for Building Applications. Determination of the Apparent Density.
11. EN 13820:2003 E. Thermal Insulating Materials for Building Applications. Determination of Organic Content.
12. EA 4/02. Expression of the Uncertainty of Measurement in Calibration. European Co-operation for Accreditation, 1999.
13. ISO 5725-2:1994 E. 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.
14. **Draper, N., Smith, G.** Applied Regression Analysis. Moscow, Finance and Statistics, 1986: 366 p. (Russian translation).
15. **Sakalauskas, V.** Statistica with Statistica. Vilnius, Publ. House “Margi raštai”, 1998: 230 p. (in Lithuanian).
16. ISO/IUPAC/AOAC International Harmonized Protocol (Written by Thompson and Wood), 1996.
17. ISO/IEC Guide 43-1:1997 E. Proficiency Testing by Interlaboratory Comparisons. Part 1. Development and Operation of Proficiency Testing Schemes.
18. **Bergonnier, S., Hild, F., Rieunier, J-B., Roux, S.** Strain Heterogeneities and Local Anisotropy in Crimped Glass Wool *Journal of Materials Science* 40 2005: pp. 5949–5954.

Presented at the National Conference "Materials Engineering'2009" (Kaunas, Lithuania, November 20, 2009)