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PROJECT REPORT

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Dissemination Level

PU	Public	
PP	Restricted to other programme participants (including the Commission Services)	
RE	Restricted to a group specified by the consortium (including the Commission Services)	
CO	Confidential, only for members of the consortium (including the Commission Services)	✓

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1. Publishable summary

1.1. Executive summary

Refractory products are materials essential for all highly industrialised processes performed at elevated temperatures such as iron- and steelmaking, cement production, glass, ceramics, nonferrous metals, power production and waste incineration. Refractories have high economic importance and serve a key function for the industry in Europe. Their technical suitability is warranted through physical, chemical and technological materials properties, laid down in material data sheets. For the purpose of obtaining those technical specifications, testing methods are described in national and international standardisation systems, like DIN, CEN or ISO. Most testing methods used for the determination of critical mechanical and physical properties of refractory products have not been thoroughly reassessed whereas in the meantime, refractory products have significantly evolved. The adequacy of current testing standards to fulfil today's market requirements has become highly questionable as for instance, current European testing standards lack any statement regarding the accuracy and precision that can be expected from the test methods described.

The ReStaR project was the first step in a systematic investigation of the accuracy, precision, reproducibility and limitations of the current European testing standards (ENs) for refractories and to enhance their relevance, especially taking into account the specific needs of the SMEs. The project contributed to improve, promote and ensure the reliability, precision and efficiency of the current European testing standards which form the base of the technical data sheets for refractory products. Keeping in mind that more than 60 % of the European refractory manufacturers are SMEs and many of them operate worldwide, the economic impact of reliable and widely recognised EN testing standards should not be underestimated.

An extensive investigation of the current EN testing methods, through designs of experiments and interlaboratory collaborative testing programme, involving the major European refractory testing laboratories, was the key approach followed. The purpose of the interlaboratory collaborative tests is to test the performance of the testing methods in the hands of the typical users. Thanks to the design of experiments and the collaborative tests the reliability of the actual European standards was assessed and their shortcomings identified and subsequently improved. Expertise gained during the whole project not only helped to strengthen the quality of the current EN testing standards, but also promote a sound methodology for the development of future standards for the refractory products.

The successful review of the EN testing standards and the effective dissemination of the project results was achieved thanks to a strong and broad-based cooperation between transnational partners. For this purpose, the PRE (only European SME-AG for the refractory industry) brought together the most active European testing laboratories and SMEs to conduct a large scale and in-depth study of EN testing standards. Under the umbrella of the PRE, the national SME-AG were involved in the project and actively participated in the dissemination of the results. Special attention was paid to address the interests of SMEs as potential users of standards.

1.2. Description of project context and objectives

ReStaR is the acronym of “Review and improvement of testing Standards for Refractory products” and brought together thirteen partners from the refractory sector in a European wide concerted R&I effort. The project was supported by funding from the “Research for the benefit of SMEs” scheme under the 7th Research Framework Programme of the European Union. This funding scheme focuses on strengthening the competitiveness of SMEs and improving industrial competitiveness across the European Union. Supported by the European Refractory Producers Federation PRE, the organisation representing the European refractory industry (Brussels, Belgium), the project ReStaR was aimed at improving, promoting and ensuring the reliability, precision and efficiency of the most important EN testing standards for refractory products.

ReStaR started on 1st October 2013 and lasted 24 months. The project was coordinated by the European Centre for Refractories (ECREF) and involves 13 partners from 7 countries (Tab. 1).

Tab. 1 - The “ReStaR” consortium

Participant	Role in the “ReStaR” consortium	Country
European Centre for Refractories (ECREF)	Coordinating body - SME	Germany
Cerame-Unie / PRE (CU / PRE)	European Refractories Producers Association – SME association	Belgium
Forschungsgemeinschaft Feuerfest e.V. (FGF)	R&D performer	Germany
Instituto Tecnológico de Materiales (ITMA)		Spain
ICAR		France
Instytut Ceramiki i Materialow Budowlanych (ICiMB)		Poland
Centre de Recherches de l’Industrie Belge de la Céramique (CRIBC)		Belgium
LUCIDEON		United Kingdom
RHI		Austria
Calderys		France
Etablissement Haasser	SME refractory producer	France
Refractaria S.A.		Spain
European Committee for Standardization	Technical Standardization Committee	Belgium

1.2.1. What are refractory products

Refractories are heat-resistant and chemical-resistant materials that form the linings of high-temperature furnaces, reactors and other processing units. They play a triple role of providing mechanical strength, protection against corrosion and thermal insulation. They need to withstand all types of stresses (mechanical, thermal and chemical) encountered at high level temperatures, such as fusion, erosion, creep deformation, corrosion and thermal shock. Refractories are materials essential for all production processes which are performed at elevated temperatures e. g. iron and steelmaking, cement production, glass, ceramics, nonferrous metals, petrochemical processing power production and waste incineration; wherever high temperatures are needed for production purposes (Fig. 1). Without refractory products there would be no cars, no planes, no trains, no petrochemical products or electricity. They have high economic importance and a key function for the industry in Europe.

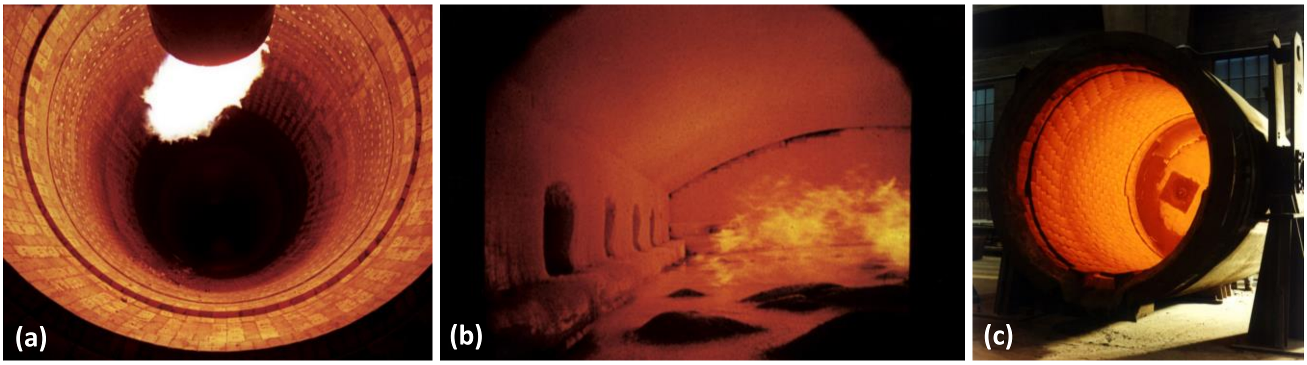


Fig. 1 - Examples of application of refractory products: lining of (a) rotary kiln for the cement industry (b) glass tanks for the glass industry (c) ladle for the steel industry (source: RHI)

In general terms refractory products are classified according to their density and the form in which they are presented. They can be "insulating" or "dense", the former are low density materials (with more than 45% total porosity), and the latter correspond to higher density (with less than 45% total porosity). According to their geometry they can be classified as "shaped", delivered in their final geometry, and "unshaped" products, delivered as a mass to be formed on site, being the most important ones of the latter refractory castables.

1.2.2. Testing Standards for refractories

The technical suitability of refractory products is evaluated and warranted through the acquisition and interpretation of physical, chemical and technological specific properties. For this purpose, testing specifications are set down in national and international standardization system, such as DIN, CEN or ISO.

Testing standards are vital for producers, installers and end-users of the refractory products. Producers need approved and recognized standards for the evaluation of their own production and the establishment of the technical data sheets, basis of their commercial argumentation. Installers rely on the standards-compliant test results to guarantee their service to end-users. Finally, end-users with testing facilities may compare their own measurement results or the performance of the products with those of the producer´s technical data sheets.

Standards tend to increase competition and allow lower products costs, benefiting economies as a whole. It has been calculated by DIN, the German standardisation body, that standardisation adds approximately 1 % to the value of manufacturing products in Europe [1].

Although EN testing standards for refractory products are well-established and accepted, their roots stretch back over 60 years and to this day, their content has only been adjusted to modern processing technology. No global and in-depth investigation of the repeatability, precision and reproducibility of EN testing standards for refractory products has been carried out for decades. Current European testing standards for refractory products show weaknesses, as the testing results are accused of lacking of accuracy/repeatability, precision and reproducibility: EN testing standards for refractories need to be consolidated and made future-proof.

The European refractory industry is aware of around 65 standards to ensure the classification and characterisation of refractory products. This project focuses on the testing standards. To avoid a far too time-consuming study and dispersion of the effort in too many directions, the scope of the project was limited to the five most commonly used testing methods. These five testing methods are typically used to measure the six widely used critical mechanical and physical properties of refractories: Bulk Density (BD), open Porosity (oPo), Cold Crushing Strength (CCS), Modulus of Rupture (MoR), Permanent Linear Change (PLC), Refractoriness under Load (RuL).

Bulk density/ open porosity:

The bulk density (BD) is the amount of refractory material within a volume (kg/m^3). An increase in bulk density of a given refractory increases its volume stability, heat capacity and resistance to slag penetration. The apparent porosity or open porosity (oPo) is the volume of the open pores, into which a liquid can penetrate, as a percentage of the total volume of the refractory. This property is important when the refractory is in contact with molten charge and slag. A low apparent porosity prevents molten material from penetrating into the refractory and therefore enhance it resistance to corrosion.

Cold Crushing Strength:

The Cold Crushing Strength (CCS) represents the ability of a product to resist failure under compressive load at room temperature. It has an indirect relevance to refractory performance and quality control, and is used as one of the indicators of abrasion resistance. The higher the CCS of a material is the greater should be the resistance to abrasion. The determination of cold crushing strength (CCS) is also highly important in case of refractory insulating bricks where bricks have to be porous as well as strong.

Modulus of Rupture:

The Modulus of Rupture (MOR) indicates the material bending strength and its suitability for use in construction. It depicts the strength of the bonding system of the refractory product, and is an important parameter for quality control and development of refractory linings. For the test a rectangular test sample is supported across a span. The testing machine applies a load at a specific rate to the centre of the sample until it breaks. The modulus of rupture is calculated using the load at which the sample failed, the span between the support, and the cross section of the sample.

Permanent Linear Change:

Permanent Linear Change (PLC) is a factor used to judge the suitability of refractories in ranges of temperature limits. Refractory materials can undergo mineralogical changes, phase transformation or shrinkage when heated. These processes may result in either volume expansion or reduction. Upon cooling to room temperature, the material will possibly be larger or smaller than the original dimensions. PLC is the property of shaped refractory to retain their original size after undergoing through a given temperature-time treatment and subsequent cooling down to room temperature. It is a crucial parameter for the design of refractory lining.

Refractoriness under Load:

The temperature range in which the softening of refractory products occurs is not identical with the melting range of the pure raw material; therefore it must be reliably determined. Refractoriness under Load

(RuL) is a measure of the deformation behaviour of refractory ceramic products subjected to a constant load and increasing temperature. The RuL is of great importance to check the suitability of refractory products for high-temperature applications; it gives an indication of the temperature at which the product will collapse, in service conditions with similar load.

1.2.3. Objectives in a nutshell

The prime scientific objectives were to:

- quantify and improve the repeatability and reproducibility of the EN testing standards for refractory products;
- highlight and optimize the practice relevant Influencing factors for the different testing methods;
- obtain reliable precision data for EN testing standards (reference sheets);
- bring the EN testing standards in line with the state-of-the-art;
- develop a robust investigation method for the systematic review of testing standards (guidelines and methodology).

The economic objectives for refractory producers/retailers were to:

- increase the competitiveness of the European SME refractory producers;
- improve the reliability of the EN testing standards;
- prevent the occurrence of off-specification batches and complaints from refractory (end-)users;
- demonstrate the quality of reviewed EN testing standards with the perspective to adopt European Standards in the ISO system;

The societal objectives were to:

- increase the involvement of SMEs in the standardization work;
- increase the international recognition of EN testing standards;
- promote European collaboration for the standardization work.

1.3. Main S&T results/foregrounds

1.3.1. Introduction to the ReStaR Achievements

The project ReStaR was characterized by an intense experimental activity. For the first time since the establishment of the EN testing standards for refractory products, the revision work for the testing standards was not only based on the diverse and sometimes contradictory experiences of the CEN Technical Committee (CEN/TC) experts or specific investigations at national level, but on an international exhaustive study on the reliability of the standards testing methods. The project did not involve only experts but also the resources of whole research facilities and SMEs, and offered a unique opportunity to confront and combine the experiences of the experts with a solid foundation of experimental results and procedures, in order to finally bring forward consistent improvements as well as establish a sound global consensus.

Initial screening experiments to identify relevant parameters for the investigated testing methods, being lead and involved only one testing laboratory, the investigations became a cross over between the different testing laboratories with the implementation of specifically designed studies (Factorial

experiments) involving at least four different RTD. Then, collaborative tests (round robin test) involving eight RTD performers and two SMEs refractory producers were carried out. For each testing method under investigation, samples from five types of refractory products (dense shaped, dense unshaped, carbon bonded shaped, heat-isolating shaped and heat-isolating unshaped) were distributed within the project partners and tested according to the better defined investigated testing standards.
Different ways were proposed to review and improve the investigated EN testing standards:

- Be more specific as regards testing parameters which are defined too vaguely in the current version of the EN testing standards, with the aim to improve repeatability and reproducibility of the testing results.
- Introducing new testing parameters, leading to an improvement of repeatability and reproducibility of the results or helping to perform more reliable measurements.
- Possibly relaxing testing parameters that are too restrictive or burdensome, if this does not or does only marginally impact the repeatability and reproducibility of the testing results. Thereby, more flexibility in the use of EN testing standards would be achieved and become more user-friendly in their use, especially for SMEs.

1.3.2. Methodology

The old piece of wisdom: “the way is the aim” apply quite well to the project. Although the final outcomes of the experimental activities are decisive results, providing crucial information on the reliability of the European testing standards (ENs) for refractory products, many of the most practice relevant propositions for improvement made for the EN testing standards are rooted in the steps undertaken and experience gained during the project. In other words, the methodology applied during the project to investigate the EN testing standards is almost an important results as the experimental outcomes themselves.

The testing standards, describing how to apply the testing methods for a given material or class of material, introduce testing parameters as well as instructions for the preparation of test specimens. These testing parameters and instructions influence, to a greater or lesser degree, the results and the repeatability of the testing methods. Overly vague definitions of these testing parameters also leads to divergent laboratory practices and fluctuation in the results, in spite of adhering to the standards.

For the sound project execution with focus on the testing methods a four step work plan was proposed and applied:

- Phase 1: Analysis of the testing method to identify the relevant influencing factors
- Phase 2: Appraisal of the influencing factors
- Phase 3: Verification of the optimized testing method
- Phase 4: Drafting of reviewed testing standards / improving the standards

which were the base for work packages.

Besides, a work package to develop and manage the tools necessary to efficiently monitor and achieve the investigation of the EN testing standards have been added. During the whole project, especially through the collaborative tests, a huge amount of data was generated. The management and the exploitation of such a database require specific and proper tools.

Finally, as is customary for projects supported by the European Union's Research and Innovation funding programme, a work package dedicated to the management activities as well as a work package for dissemination activities were implemented in the work plan.

The implemented methodology is therefore quite linear, building on the results achieved during the preceding step(s).

1.3.2.1. Phase 1: Analysis of the testing method to identify the relevant influencing factors

The first phase of the project was dedicated to a systematic and comprehensive investigation of the potential testing parameters or factors that can influence the repeatability, reliability and efficiency of each of the testing standards under examination.

To efficiently conduct these extensive studies, one laboratory was responsible for one testing method. This laboratory was selected according to its experience, testing possibilities and capacities as regards to a given testing method. Using various designs of experiments (e.g. screening factorial designs) and statistical evaluation, influencing testing parameters were identified and, at the scale of one laboratory, quantified. Although basically performing the practical investigation alone, the laboratory responsible for a testing method also relied on the experience from the other project partners. To this end, meetings and conference calls promoted the exchange of information and experience. Most of the influencing factors were known but their influence is often over- or underestimated.

In this first phase, the laboratory responsible for a testing method enjoyed a large degree of freedom to investigate the testing method in question. Even exotic potential influencing factors were investigated, "negative results" are results as well. Furthermore, comparison with testing standards in other international standardisation systems (ASTM, JIS, etc) revealed differences and additional or unspecified information. These differences proved to be particularly informative, once comprehended and appropriately "exploited", as potential sources of improvement.

The different RTD Performers responsible for the testing methods implemented slightly different investigation strategies, using screening factorial designs for some of them, others applying full factorial designs. Especially since the cost and time for performing one measurement considerably varies from one testing method to another testing method, from less than 15 min to more than one day for one test piece, investigation plans had to be adapted. In addition, depending on the testing method under review, different amount of potential influencing factors could were identified and investigated, also promoting different investigation strategies.

Finally, some intuitive effects were experimentally confirmed. Other testing parameters were found to have much less impact than expected. Finally even counter-intuitive effects were brought to light. More specific details regarding the investigated testing methods are given in the relevant part of this report (session *1.3.3 Findings per testing methods and proposed improvements to the relevant EN testing standard(s)*).

1.3.2.2. Phase 2: Appraisal of the influencing factors

In a second phase, the impact of the testing parameters or factors, identified in phase 1, were quantified at a much larger scale. For each testing method under investigation, specifically designed studies (Factorial experiments) involving at least four different laboratories were set up and performed. A factorial experiment is a strategy to draw conclusions about more than one factor, or variable in an astute and time-saving manner. Factors are thereby deliberately simultaneously varied, instead of one at a time. Using statistical laws, an efficient evaluation of the effects and possible interactions of the influencing testing parameters are achieved. In addition, data relating to the reproducibility of the investigated methods for different kinds of refractory materials were assessed.

Although factorial experiments are much more efficient than studying one factor at a time, it remains relatively labour and time intensive if a large number of factors are considered. Hence only a limited number of factors, identified in the phase 1 as relevant in practice and presenting consistent potential for improvement, were to be considered for the phase 2.

The objective of this phase was to measure the real impact of the influencing factors, and to pick out the factor values which are the most favorable for the practice and/or conduct to the best reproducibility. For a given operator or even laboratory, by force of repetition and establishment of internal testing routine over the time, a relative good *repeatability* may be reached. However, because of vaguely or suboptimal defined influencing factors (a too wide range of value, or different sample geometry) even a given testing standard, may lead to different internal testing routines between different laboratories. The direct consequence is poor *reproducibility* by applying the testing standard.

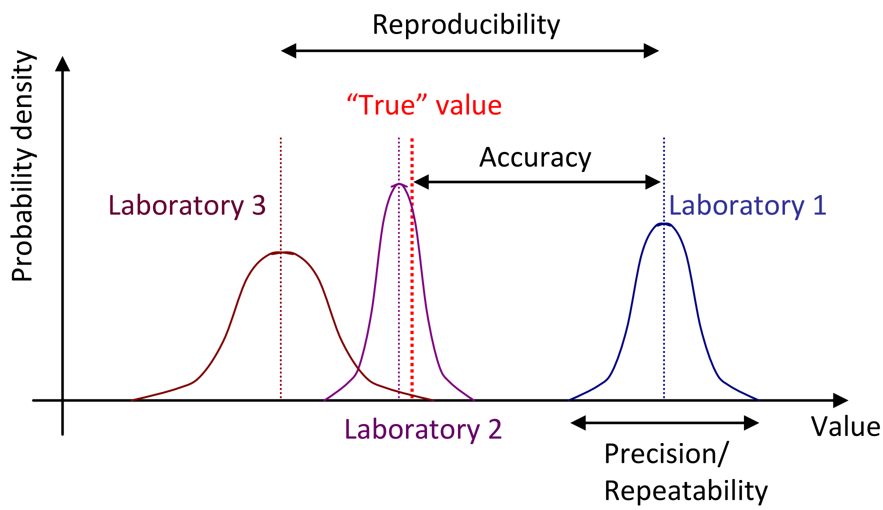


Fig. 2 - Graphical illustration of the concept of accuracy, precision/repeatability and reproducibility

At the end of this phase, first reliable findings on the *repeatability* and *reproducibility* were obtained. Further, a good understanding of the impact of the most relevant testing parameters was achieved and first proposals for the improvement and review of the testing standards formulated.

A significant issue at this step for the project ReStaR was the preparation of the test specimens: should they all be prepared by one partner in order to focus on the testing method itself? or should each partner prepare their "own" test specimens since, in practice, users of the testing standards have to systematically prepare the test specimens themselves. Additionaly, in order to exclude the impact of fluctuations in raw material quality or production process on the material properties, the refractory products used for the investigations came from a single batch of production. At this stage of the project, it was decided that each partner prepares their "own" test specimens as part of the testing process while using the testing standard.

1.3.2.3. Phase 3: Checkout of the optimized testing method

The third phase aimed at putting to the test the conclusions of the phase 2 and checking the reliability of the reviewed testing standards. Almost even more important, repeatability and reproducibility figures (precision data) for each testing method were gained during this phase. These precision data are to be included in the reviewed EN testing standards. To this end, a round robin testing programme was established and the data collected were statistically processed using the Analysis of Variance (ANOVA) random effects model.

For the analysis of interlaboratory test results, the user-friendly commercial solutions (software) PROLab from Quodata was used. PROLab implement directly the international standards ISO 5725. The monitoring of interlaboratory studies is challenging and arduous process. Depending on the testing method, even a single test may be time-consuming. The testing procedure and the test pieces preparation were carefully documented in order to understand suspect/surprising results. An ongoing exchange between the involved laboratories and the interlaboratory study leading institution ensured tracking of progress, identification of difficulties and the implementation of remedial (or corrective) actions.

Interlaboratory studies (round robin tests) involving eight RTD performers and two SMEs refractory producers were carried out. For each testing method under investigation, samples from five types of refractory product (dense shaped, dense unshaped, carbon bonded shaped, heat-isolating shaped and heat-isolating unshaped) were distributed amongst the project partners and tested according to the better defined investigated testing standards.

During the third phase of the project ReStaR, a "hybrid" solution concerning the test specimen preparation was implemented. The first steps of the test specimen preparation were undertaken by a limited number of laboratories, usually two different laboratories for one combination testing method / type of refractory product. The final steps of the preparation were then managed by the laboratory that would finally perform the test according to the reviewed testing standard. That means for one combination testing method / kind of refractory products usually two different "populations" of test specimens were tested: for instance, first preparation steps achieved by lab1 then final test specimen preparation performed by lab1 to lab5 and first preparation steps achieved by lab6 then final test specimen preparation performed by lab6 to lab10. Therefore, the impact of the preparation, at least first steps of the preparation, could, to a certain extent, had been investigated (i.e. significantly different test results as well as significantly difference for *repeatability* and *reproducibility* of the two different "populations"?).

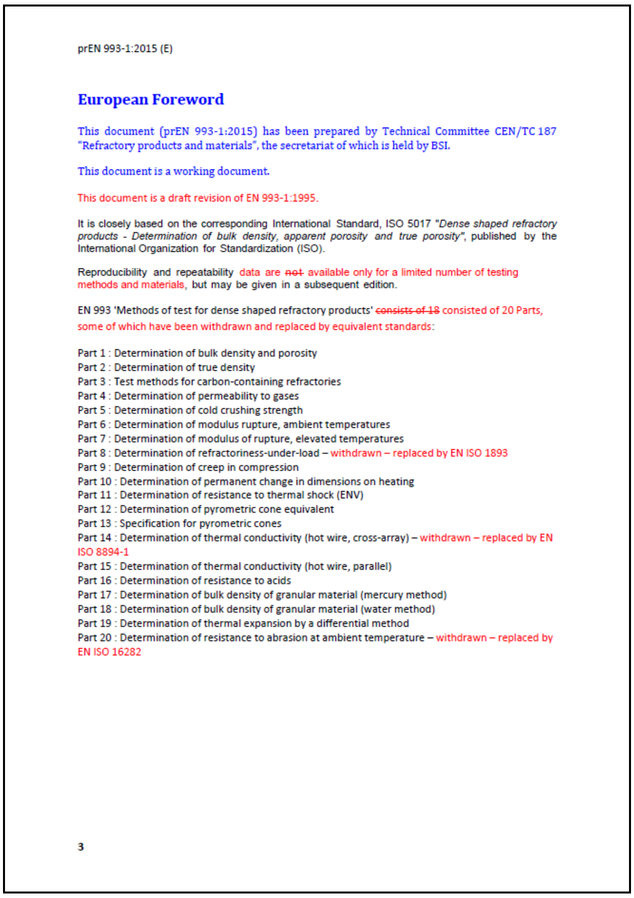
The ReStaR project consortium adopted a rather conservative position for the planning of the interlaboratory studies. Most of improvement propositions implemented for the interlaboratory studies correspond therefore to a better specification of the standard. Testing parameters that could be potential loosened according to the results of the previous investigations/phases, were set at values corresponding to the current testing standards for the interlaboratory studies. Thus, even if not all the revision propositions for the testing methods would be implemented in the future version of the EN testing standard for refractory products, the precision data generated during the phase 3 of the project are still relevant and could be integrated anyway in the EN testing standards.

1.3.2.4. Drafting of reviewed testing standards / improving the standards

The final phase of the applied methodology deal, of course, with the drafting of reviewed testing standards building on the results and expertise gained during the preceding phases. Revision of testing standards at the European level were initiated by the CEN technical committee (CEN/TC), working group consisting of experts in the field of refractory testing. Any amendments to the current standards have to be discussed in CEN/TC meetings. The proposed methodology offers the opportunity to confront and combine the experiences of the experts with a solid foundation of test results and procedures, and thus to establish a sound global consensus.

The ReStaR project itself finds its roots in a CEN/CT 187 meeting. Most of the experts from the CEN/TC 187 and their institution/company, as well as the CEN/CT187 chairman, were therefore involved in the ReStaR project.

Drafts of the reviewed testing standard were at first managed by the RTD performers responsible for one testing method during the preceding phases of the project. Using all their accumulated experience, the concerned RTD performers issued proposition for improvements. These improvements took different forms but basically aiming at facilitating the use of the standard through for instance better guiding and informing the user of the standards and reducing the interpretation and potential causes of discrepancies between users. A first discussion on the issued propositions took place during a project meeting six months before the end of the project. The drafts including these propositions for improvements were then worked out and dispatched to the project consortium members for comment. Finally intensive and extensive discussions of the drafts took place during the project final meeting to conclude with a formal approval on the proposals/drafts (Fig. 3) to be put forward to the CEN/TC 187 for refractory products and material by the project consortium by the end of the project.



prEN 993-1:2015 (E)

European Foreword

This document (prEN 993-1:2015) has been prepared by Technical Committee CEN/TC 187 "Refractory products and materials", the secretariat of which is held by BSI.

This document is a working document.

This document is a draft revision of EN 993-1:1995.

It is closely based on the corresponding International Standard, ISO 5017 "*Dense shaped refractory products - Determination of bulk density, apparent porosity and true porosity*", published by the International Organization for Standardization (ISO).

Reproducibility and repeatability data are ~~not~~ available only for a limited number of testing methods and materials, but may be given in a subsequent edition.

EN 993 'Methods of test for dense shaped refractory products' ~~consists of 18~~ consisted of 20 Parts, some of which have been withdrawn and replaced by equivalent standards:

Part 1 : Determination of bulk density and porosity
Part 2 : Determination of true density
Part 3 : Test methods for carbon-containing refractories
Part 4 : Determination of permeability to gases
Part 5 : Determination of cold crushing strength
Part 6 : Determination of modulus rupture, ambient temperatures
Part 7 : Determination of modulus of rupture, elevated temperatures
Part 8 : Determination of refractoriness-under-load – withdrawn – replaced by EN ISO 1893
Part 9 : Determination of creep in compression
Part 10 : Determination of permanent change in dimensions on heating
Part 11 : Determination of resistance to thermal shock (ENV)
Part 12 : Determination of pyrometric cone equivalent
Part 13 : Specification for pyrometric cones
Part 14 : Determination of thermal conductivity (hot wire, cross-array) – withdrawn – replaced by EN ISO 8894-1
Part 15 : Determination of thermal conductivity (hot wire, parallel)
Part 16 : Determination of resistance to acids
Part 17 : Determination of bulk density of granular material (mercury method)
Part 18 : Determination of bulk density of granular material (water method)
Part 19 : Determination of thermal expansion by a differential method
Part 20 : Determination of resistance to abrasion at ambient temperature – withdrawn – replaced by EN ISO 16282

3

Fig. 3 - Foreword from the draft standard EN 993-1 with proposed modification in red

1.3.3. Findings per testing methods and proposed improvements to the relevant EN testing standard(s)

In this specific session of the report, more specific details regarding the investigated testing methods and related improvements to the EN testing standards are given.

1.3.3.1. Bulk density (BD) / open porosity (oPo)

The bulk density (BD) is the ratio of weight (or mass) to volume and is expressed in kilograms per cubic metre (kg/m³). An increase in bulk density of a given refractory increases its volume stability, heat capacity and resistance to slag penetration. The apparent porosity or open porosity (oPo) corresponds to the volume fraction of open pores present in the refractory and is reported in percentage (%). A low apparent porosity prevents molten material from penetrating into the refractory and therefore enhance it resistance to corrosion. Generally, refractories with a high amount of porosity have better insulating properties but a lower density which leads to a lower corrosion resistance and a lower strength. A lower porosity can decrease the thermal shock resistance and in more porous bricks, the pores present can act as crack inhibitors. As a result, the amount of porosity and of density are critical properties to be assessed in order to use the material for its right specific service condition.

Several methods can be employed to measure the BD and oPo of materials, the so-called imbibition method is the most commonly used in Europe for quality control and described in the standard EN 993-1 for dense shaped refractory. The BD and oPo are there determined by weighing the mass of dry test sample, then its apparent mass when immersed in a liquid which it has been impregnated under vacuum, and then it's mass in air while still soaked with liquid.

The EN 993-1 for dense shaped refractory and EN 1402-6 for unshaped refractory standards were issued in 1995 and are based on the PRE/R9 and PRE/R10 (PRE Recommendation) written in 1966. The EN 1402-6 was first issued in 1998 and revised in 2003 and is based on the PRE/R28 written in 1977. Finally the EN 1402-6 was roughly accepted as an ISO (EN ISO 1927-6) in 2012 for unshaped refractory, unfortunately a number of amendments as part of a compromise had to be found with international partners these are now suspected as compromising the reliability of the test.

Investigated testing parameters and summary of the method specific investigations

The bulk density and open porosity testing methods on refractory materials presents more than 15 variables/parameters that need to be fixed along the testing procedure (Fig. 2). The setting of theses variables/parameters at "different levels", even in a range accepted by the EN testing standard, is highly suspected to influence the bulk density and open porosity results, as well as affecting the accuracy, the precision and the reproducibility of the testing results.

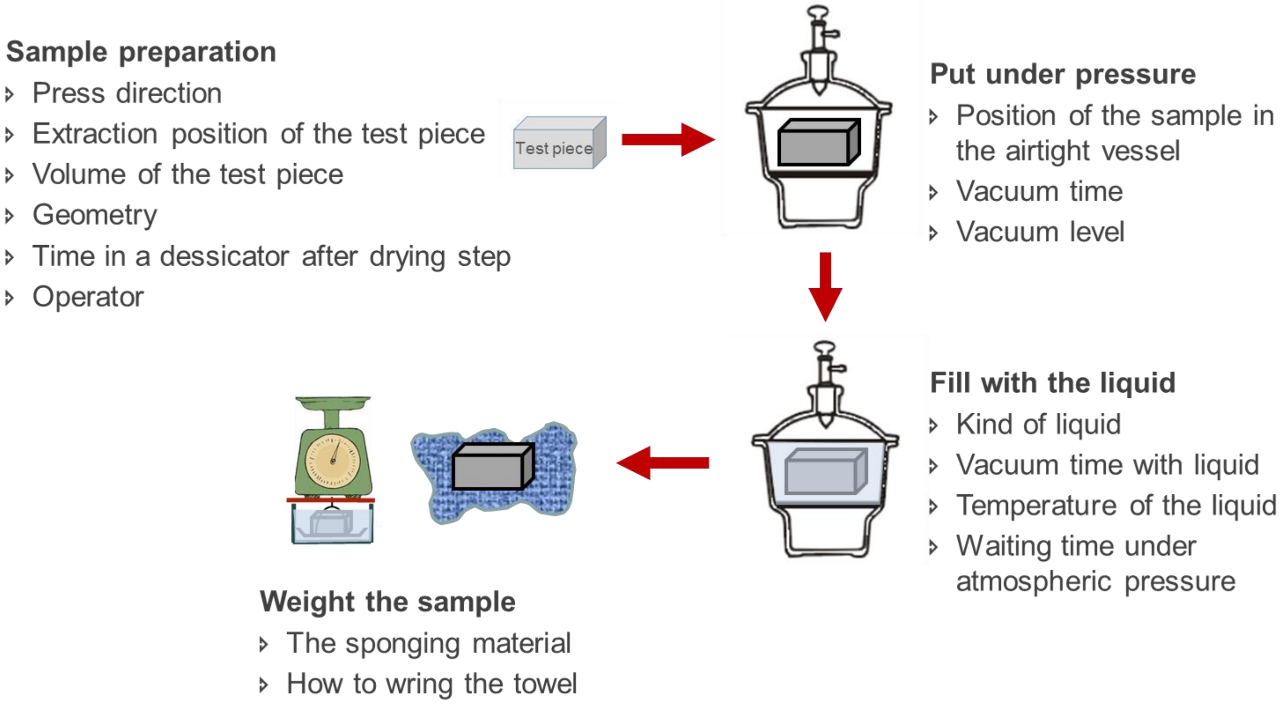


Fig. 4 - Schematic representation of BD/oPo testing procedure including a listing the different parameters/factors that may influence the testing results

An initial large investigation of these parameters/factors was performed during the first phase of the project and, in addition, permitted parameters presenting more potential for improvements to focus upon during the subsequent phases of the project. The conventional method involves changing one parameter at a time while keeping all other constant. This method may be very expensive and time consuming. In addition, it fails to determine the combined effect of different factors. So the Plackett-Burman design (PBD) was used for this first step of the study. It is the most frequently used screening design mainly due to its ability to estimate all main effects with the same precision. Furthermore a fractional factorial design present the advantage of optimizing the number experimental measurements considering a large number of variables/parameters (factors).

The study of the screening factorial design highlights the most important factors for the two standards. These factors were then evaluated with another factorial design involving five different laboratories in order to get a better knowledge of their influence on the bulk density and open porosity results and achieve an initial estimation of the precision data. Furthermore after this first step, first conclusions were formulated to improve the existing standard. For example it has been found that:

- The waiting time in a desiccator of the test piece after to be dried in the drying chamber at 110 °C in order to cool down (between 2 hours and 24 hours) has no significant impact on the results. In the standard it states that the test pieces are placed in a desiccator until it has cooled to room temperature. But in practice, in laboratories, the actual time can be difficult to control. The necessary time to cool down can differ from 30 min to more than 2 hours, which depends of the material and its volume. As no significant impact on the results were noticed, in order to save time, the samples can be put in the desiccator at the end of the day before to be directly tested the next morning for example.
- The position of the samples in the airtight vessel has no significant impact on the results. The samples are enough small and the airtight vessel enough big to have at least two or three levels of samples in the vessel. So it means that the whole vessel with several position levels of test pieces can be used to study the bulk density and the open porosity without significant effect. So time and money can be saved.

During the second phase of the project, the four factors presenting most potential for improvements identified during the screening factorial design (phase 1) were then investigated in three different design of experiments. Each design of experiment involved five laboratories and was applied to one representative and/or prominent refractory products for a given standard:

a) Material HA75 high alumina product (EN 993-1: For dense shaped refractory)

The time of maintaining the vacuum pressure (Tvac), the kind of sponging material (Sp), the ratio of the longest to the shortest dimension of the test piece (L/d) and the time that the sample is under the immersion liquid after to have removed the vacuum pressure (Wwp) were the four studied influencing factors for this refractory product.

b) MC95/10 magnesia-carbon refractory product (EN 993-1: For dense shaped refractory)

The time of maintaining the vacuum pressure (Tvac), the kind of sponging material (Sp), the geometry of the test piece (Geo) and the vacuum pressure (Vac) were the four studied influencing factors for this refractory product.

c) dense refractory castable MCC75 (EN ISO 1927-6 for the unshaped refractory)

The format of the test piece (For), the kind of sponging material (Sp), the kind of immersion liquid (Liq) and the vacuum pressure (Vac) are the four studied influencing factors for this refractory product.

For each design of experiment and accordingly kind of refractory product, the results obtained from the different laboratories (i.e. 5 factorial designs) were statistically analysed using the software Optival in order to determine for each laboratory the influencing factors. Furthermore an evaluation of the ring test was performed with the software PROLab, to obtain information on the repeatability and the reproducibility of each run of the factorial design.

Finally, an interlaboratory test (round robin test) was performed involving 10 laboratories for the three previously mentioned refractory products. Thereby, the most suitable combination of testing parameters from a practical perspective (“user-friendly”) and giving satisfactory repeatability and reproducibility during the second phase of project was carefully selected. Precision data, currently missing from the EN testing standards, for the three investigated refractory products were generated as guidance values for the EN testing standard user.

Proposition of improvements for the EN standard & implications

Thanks to the whole study on the bulk density and open porosity testing standard EN 993-1 for dense shaped refractory, new recommendations to improve this standard were formulated. Some recommendations can simplify the testing procedure for the SME without affecting the precision data and some can increase the repeatability and the reproducibility of the testing results. Precision data derivedfrom the round-robin test have been also added in the recommendations. As examples:

- For the determination of density of immersion liquid, it is possible for a question of simplification of the calculation, to assume that 1 cm³ of water weights 1 g at room temperature. This simplification leads to a slight overestimation of the bulk density results, which remains however clearly smaller than the repeatability of the testing method.
- A special note was written concerning the immersion liquid. The use of paraffin as immersion liquid is often uncomfortable and more expensive. Furthermore, the different commercial paraffins offer a large range of viscosity and density, leading to different degrees of infiltration of the tested test piece, which in turns greatly affect the reproducibility of the testing method in comparison with the use of water.
- It is recommended to use a linen cloth or a sponging towel as sponging material for the test. If a leather towel is used, a lower reliability in the testing results is expected
- If water is used as immersion liquid it is recommended to wait until the water boils to ensure the maximum vacuum is attained for the test temperature.

1.3.3.2. Cold Crushing Strength

One of the most important parameters characterizing refractory products is the cold crushing strength (CCS), which is practically universally supplied in refractory products datasheets and is a prerequisite for the technical acceptance of products. The accuracy of information on data sheets is vital as they are used by refractory producers and customers as a common basis for negotiation whether refractory products are suitable for their respective requirements and applications. End-users with testing facilities may compare their own measurement results to the technical data sheets. It is therefore important to use a standardised method for determining the CCS with satisfactory precision. This is particularly relevant for SMEs, where unreliability by CCS determination may seriously affect the finances and position of the enterprise.

Cold crushing strength is determined at uniaxial compression test at ambient temperature and is calculated from the maximum force that test piece can withstand before failure occurs divided by the area over which the load is applied.

Refractory products as brittle and inhomogeneous materials vary from one specimen to another. Moreover, the preparation of test pieces and the testing conditions can influence the test results and their dispersion. Different standards describe procedures for CCS testing for dense-shaped refractories, heat insulating products and unshaped products (Tab. 2).

Tab. 2 - Standards for CCS of refractory materials tests

Dense shaped refractory products	Shaped insulating refractory products	Monolithic (unshaped) refractory products
EN 993-5 ASTM C 133 ISO 10059-1 ISO 10059-2	EN ISO 8895	EN ISO 1927-6

According to the existing standards for each group of materials, different requirements apply to the shape, size and dimensional tolerance, loading rate, hardness and roughness of the loading plates and packing. The above requirements are not fully defined in all of the standards. There are also parameters, which are not described in the testing standards e.g. the casting direction and grinding for unshaped products. As a result, the influencing testing parameters related to the CCS test may vary in different laboratories and affect the obtained results.

Investigated testing parameters

The following factors were investigated for dense shaped refractory products:

- **location of test piece extraction**. In practice differences between CCS results for test specimens from the middle of brick and from the corner due to inhomogeneity of material are common. It is important to compare influence of this factor to the other factors influencing CCS results.
- **Geomtrical factors: shape, height and size** are related to the volume of tested sample. Brittle refractory products with porous texture characterise dispersion of strength. Therefore, it was expected that obtained results of CCS tests depends also on those test pieces parameters.
- **smoothness of the loaded surfaces**. It is an important factor because if the loaded surface of test piece is flat and smooth the load distributes uniformly on whole surface.
- **deviation of parallelism and perpendicularity of the test pieces**. Lack of parallelism and perpendicularity of test pieces can introduce local stresses and irregularity of loaded surface cause decrease of real loaded area. In all cases the CCS results will be reduced.
- **load orientation, load rate and preload**. Deviation of CCS of materials depending on the load direction relative to direction of formation can be observed as result of textural differences and load rate is related to speed of crack propagation
- **hardness and roughness deviation of applied steel plates**. Roughness can cause differences in stresses distribution during test and it may change in cyclical application depending on hardness of steel. It was important to investigate whether that differences influence the results.
- **packing** (with/ without). In ASTM C133 and ISO 10059-2 the packing (cellulose fibre wall board) is applied. The aim of packing application was to obtain more uniform pressure on cross section of test piece and alignment of uniformity on surface. However, A. Majdič et al. [2] showed that packing led to reducing the CCS values of specimens with ground surfaces.

For shaped heat insulating materials the tested factors were as follows:

- **The load rate and preload.** There are two different load rates (0,05 ± 0,005 MPa/s and 0,2 ± 0,02 MPa/s) applied in EN standard. It is important to check the influence of chosen load rate on CCS results. ASTM C 133 suggested special attention to ensure accurate adjustment of device and using small initial load before CCS tests
- **Dimensions of test pieces.** The dimension of test piece and accordingly the loaded surface on dispersion of results is expected influence the testing results.
- **Packing**
- **Operator**

The following factors chosen for investigations of unshaped materials:

- **Size of the test piece.** The size of test pieces allowed in standards significantly vary: three different bar formats (bars) are proposed
- **Method of test piece preparation.** According to existing standard tested piece may be a cube cut from the bars or the half of the tested bar after determination of the modulus of rupture.

Cast direction. If the sample is prepared in laboratory before be tested the cast direction is known. But in case of testing of item already casted by a third party, it may prove difficult to establish the cast direction. Therefore, it is important to evaluate the influence of this factor on CCS results.

- **Load rate** and preload.
- **Grinding.** The loaded surface should be flat and smooth and it is important to know whether additional grinding is necessary in case of cast sample.
- **Packing**.

Summary of the testing method specific investigations

Three groups of materials were tested:

- dense shaped refractory materials with a true porosity below 45 %: Three types of dense shaped bricks (A and B: **HA75** High Alumina >75 % Al_2O_3; C: **MC90** Magnesia Carbon >90 % MgO) were used. To limit the influence on CCS results of structural defects (heterogeneity, internal cracks and voids) within the tested materials, all bricks and test pieces prepared for CCS testing were checked by measuring the ultrasound transit time through the sample and then it was counted on ultrasound velocity.
- shaped insulating refractory materials with a true porosity of not less than 45 %: insulating bricks with height 64 and 76mm during the phase 1 and 2 of project. For the interlaboratory tests, Lightweight Insulating >35 % Al_2O_3 (**LWI35**) were used. Brick homogeneity was checked by measurement of geometric density for every prepared test piece.
- unshaped refractory products (dense and insulating): **MCC75** Medium Cement Castable >75 % Al_2O_3 as a dense unshaped refractory products was selected. To characterize tested material the content of moisture and grain size distribution was determined with according to EN ISO 1927-3. The castable was prepared according to improved instructions from EN ISO 1927-5. Before preparing the bars to CCS tests, on every single bag the consistency of castable was determined with accordance to EN ISO 1927-4. The homogeneity of bars prepared from unshaped refractory product was tested by measuring ultrasound velocity. LW 124 Lightweight castable was used as insulating unshaped refractory products for the investigation.

Special attention was paid to sampling to be sure that all tested samples are representative and to minimize the influence of material heterogeneity. Especially for inter laboratory test during phase 3 of the project, sampling was described and documented in specific document and templates.

The design of experiments and variance analysis were applied to determine the most significant testing parameters (factors), which affect the accuracy and precision of CCS testing. Plackett-Burman design was used because of its ability to estimate all main effects with the same precision and possibility of minimizing the number of experiments. The main effect of each response is evaluated as the difference between averages of measurements made at the high level and low level of each factor. The calculation of the significance of the impact of individual factors that influence the value of the CCS was extended by analysis of variance ANOVA using statistical software version V.3.3.2.4 OptiVAL from the Quodata.

Specific findings and improvements of standards

The CCS results obtained by all research performers were consistent and allowed to draw conclusions which are basis for revision of existing CCS standards and its improvements.

One of the most important finding was that the laboratory performing the measurement appeared as an important factor during CCS design of experiments. This can be explained by simultaneous influence of large number of influencing testing parameters which is connected but not fully described in the existing standards and individual laboratory practise (customs). It highlights the necessity for improvements of the existing standards and tightening of measurement procedures as a whole to ensure the measurement of much more comparable results and minimise discrepancies between different laboratories. It is important that experimental procedures described in standards will guide the user in some choices and reduce the interpretation of the recommendations included in the standard.

Besides the main conclusion the more specific findings suggest that special care has to be done during the preparation of the samples. It should be done by taking into account the most important findings resulting from ReStaR project:

1. Operator has no influence on CCS results,
2. Use packing has strong negative effect and increases the dispersion of the CCS results,
3. Load rate has influence on results in case of all tested kinds of materials,
4. The roughness of the plates in tested range has no significant effect on the results,

Additionally for dense shaped materials:

5. Shape of the samples have a significant effect on observed CCS results,
6. Influence on results of place of test piece extraction, corner or middle of the brick were noticed,
7. A statistically significant effect can be observed if the test piece is extracted parallel or perpendicularly to the press direction,
8. The test pieces surfaces should be ground.

For unshaped materials:

9. The heterogeneity of test piece due to differences in castable and test piece preparations before tests are most influencing factor,
10. Test pieces preparation by three point bending is recommended,
11. Less dispersion in the results for the Format B were obtained,
12. Grinding has negligible effect on CCS of unshaped materials and its use is not substantiated,

13. For heat insulating shaped materials there is no necessity of changing experimental procedure only small ammendments were proposed.

14. Repeatability and reproducibility data for CCS measurements for each tested group of materials were determined and should be stated in revised standards.

On the base of obtained results the revised drafts of existing EN standards were prepared. Definitions have been complemented and clarified. In EN 993-5 standard for dense refractory number of test pieces was increased from 1 to 5 in order to enable calculation of the average and dispersion of results. In the standard for unshaped refractory material tests the used apparatus was specified and described. Additional information to be reported (mean value and standard deviation for each sample and item) was introduced to the test reports contents. Precision data were incorporated to standards.

All proposed changes should facilitate the application of standards and reduce the discrepancies between users which are particularly important for SMEs.

1.3.3.3. Modulus of Rupture:

In service, depending on the stress conditions, a refractory product can fail either in compression or in tension. Tensile stresses often result from thermal gradients in products, in particular those subject to thermal cycling. Accordingly, the resistance to mechanical failure in both stress states has to be accurately determined in order to warrant the safe operation of the installations. Whereas the cold crushing strength test (CCS) allows the direct evaluation of the compressive strength of a refractory product, the tensile strength is for its part evaluated through the determination of the modulus of rupture. Strictly speaking, the modulus of rupture (MOR) is the maximum stress that a test piece can withstand when loaded in bending.

The current version of the EN standard addressing the determination of this characteristic for shaped products, EN 993-6, dates back to 1995 and is based on a three-point bending test configuration in which a prismatic test piece of given geometry is loaded at constant stress rate until failure occurs (Fig. 5). The equivalent document for unshaped products, EN ISO 1927-6, although more recent (2012) relies on the same principle and incorporates almost identical requirements in terms of test parameters.

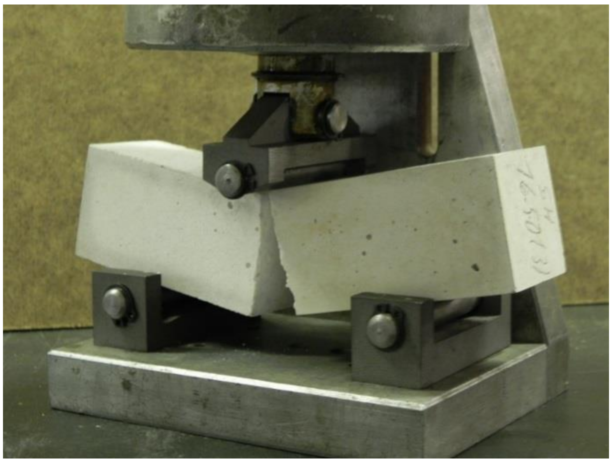


Fig. 5 - Three-point bending test according to EN ISO 1927-6

Current version of both standards incorporate primarily requirements in terms of test bar geometry, test jig design (in particular span length) and loading rate. Each of these parameters together with the prescribed tolerances have been considered as factors susceptible to influence the MOR testing results and accordingly thoroughly screened during Phase 1 and Phase 2 of this project through dedicated factorial design of experiments and subsequent statistical analysis. The rationale behind the choice has been to assess whether all current parameters are correctly described/specified and whether the recommended values thereof still fit the current products on the market. Depending the case, other potential influencing factors have been considered as for instance the nature of the surface tested (as fired vs. bulk, as cast vs bulk) or the location of the test bar within the brick. These different factors are graphically summarized in the Figure below.

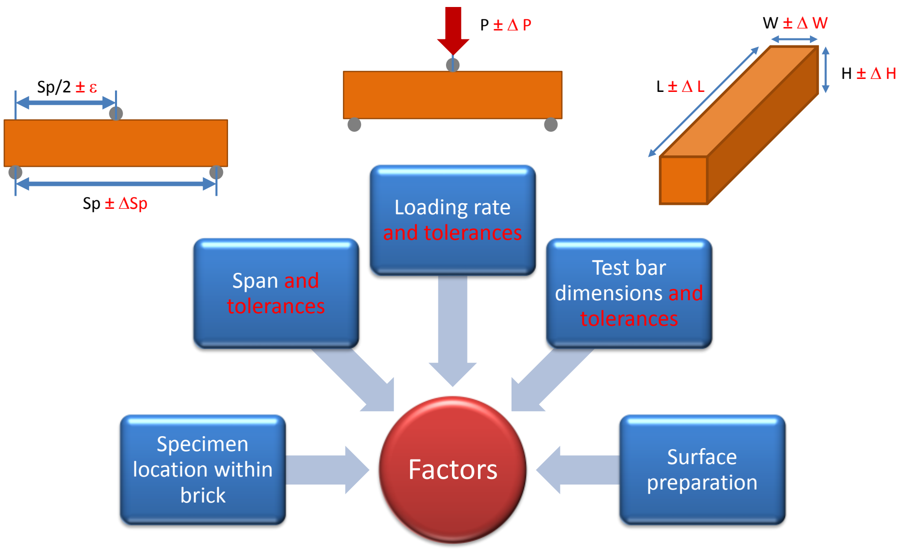


Fig. 6 - Investigated testing parameters (factors)

Overall, the selection of factors was done keeping in mind that influencing factors need to be optimized but that drastic changes should be avoided to minimize the impact on SMEs already employing the existing standards in their daily practice (e.g. the measurement principle has been kept unchanged). It has also been chosen to rely whenever possible on full factorial designs in order to avoid confounding of main effects and interactions and to run as many replicates as possible taking into account the amount of material available, in order to quantify the random error due to inevitable uncontrolled factor(s) like the product intrinsic heterogeneity for example.

Summary of the method specific investigations and findings

In Phase 1 of the project, aiming at screening the potential influencing factors, three materials have been considered : the high alumina dense shaped product specifically provided by RHI AG for the ReStaR project, a lightweight insulating shaped refractory from RATH and the medium cement castable produced by Calderys in the framework of this collaborative EU-project. More than 150 tests have been conducted on those materials from which it could be concluded that

- the geometry of the test specimen had, depending on the material, a more or less significant impact;
- the loading mode and rate had apparently no statistically significant effect;
- the influence of the surface quality and preparation was unclear and remained to be confirmed.

The same three materials have been used in the interlaboratory exercise of Phase 2. Around 300 tests have been performed by the 4 to 5 laboratories involved and have confirmed previous observations. Thereupon it has been shown that in the case of the shaped insulating product, not only the geometry but also the nature of the surface in tension influenced the outcome of the test.

Finally, this interlaboratory exercise also enabled to identify potential optimum set of conditions with regard to repeatability and reproducibility, an example of which is given in the graph below (Fig. 7).

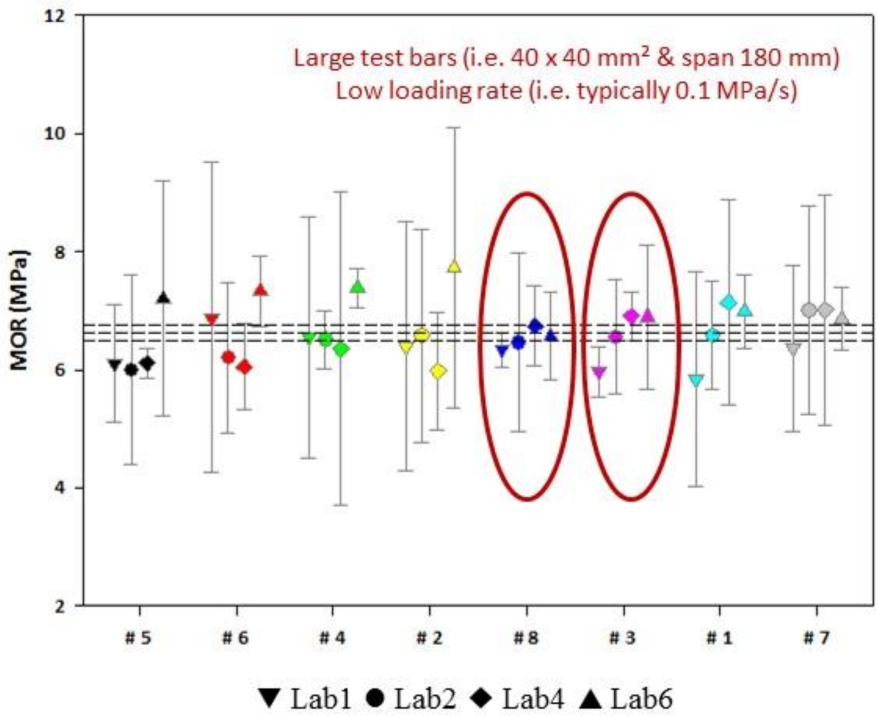


Fig. 7 - Graphical representation of the interlaboratory results for large test bars and low loading rate

These optimum sets of conditions have been further evaluated during the large scale round robin carried out on five different representative refractory materials and involving all partners. The main outcomes of this last Phase can be summarized as follows:

- in the case of shaped products, the analysis of the whole set of results indicates a statistically significant effect of the testing laboratory, specimen position within the brick and cross section (the smaller the cross section the higher the MOR) confirming previous observations;
  - the effect of the specimen location within the brick can be related to the presence on some of the specimens of surface layer with a clear different aspect, or to observed slight variation of the density within the brick ; it follows that recommendations with respect to sampling will have to be incorporated into the revised version of the standards ;
  - thereupon, the measured relative repeatability and relative reproducibility on large cross section specimens are slightly better than those achieved for small cross section specimens what can be most probably be attributed to the intrinsic heterogeneous structure of the product ; hence, increasing the tested volume, although susceptible to result in slightly lower MOR values, may have a levelling effect with respect to specimen to specimen variation ;

- for castables, the main factor affecting the results is the testing laboratory ; a twofold difference is observed between relative repeatability and relative reproducibility emphasizing the major effect of specimen preparation and the need to tighten the preparation protocol in forthcoming version of the standard ;

whichever material is considered, the loading rate does not affect the MOR value obtained ; this clearly suggests that the current tolerances on this parameter, much smaller than those adopted in this research, is satisfactory but could eventually be increased without significant impact on the MOR test results.

Proposition of improvements for the EN standard & implications

On the base of the project outcome but also after careful reading of current standards, a series of improvements have been considered and drafted in potential revised versions of the documents. These improvements aim at

- clarifying the purpose of the test e.g. by incorporating an informative paragraph highlighting the significance and use of the method
- guiding the user in some choices e.g. by providing some guidelines in the choice of the test specimen dimensions ; current standard allows indeed the use of different test bar geometries and raising the awareness of the potential user about the effect of specimen geometry is fully relevant ; following note is one example how this could be achieved:

NOTE The relative ratio of the largest grain size to the smallest specimen dimension may significantly affect the obtained modulus of rupture value. Testing specimens cut from larger items and containing large grains may result in MOR values significantly different from those obtained when testing the items from which they were cut. The smallest test specimen dimension should at least be five times larger than the maximum grain dimension.

- facilitate the use of the standard e.g. by specifying the loading rate in [N/s] thereby avoiding potential errors when converting the recommended stress rate for each different geometry in practical loading rates to be used (see the Table below taken form the proposed revised version)

8.8 *Select a loading rate depending of the material tested according to the requirements of Tab.2.*

Table 2 — Standard loading rates

Refractory type	*Dimensions (mm)*	*Span (mm)*	*Stress rate (MPa/s)*	*Loading rate (N/s)*
Dense shaped refractory	*230 x 114 x 76*	*180*	*0,15 ± 0,015*	*366 ± 37*
	230 x 114 x 64	*180*		*259 ± 26*
	200 x 40 x 40	*180*		*36 ± 3,6*
	150 x 25 x 25	*125*		*13 ± 1,3*
Shaped insulating refractory	*230 x 114 x 76*	*180*	*0,05 ± 0,005*	*122 ± 12*
	230 x 114 x 64	*180*		*86 ± 8,6*
	200 x 40 x 40	*180*		*12 ± 1,2*
	150 x 25 x 25	*125*		*4 ± 0,4*

- reducing the interpretation and potential causes of discrepancies between users e.g. by specifying a clear rejection criterion to discard questionable results in the case of fracture occurring “far” from the midpoint of the test specimen (see below)

8.11 Inspect the test specimen. If the onset of failure is located outside the range [midpoint - 5% of the Span, midpoint + 5% of the Span], discard the result of the test and repeat with a new test specimen.

- and finally incorporating the generated precision data that were missing up until now.

1.3.3.4. Permanent Linear Change:

For refractory products a key physical property is their permanent linear change (PLC). This is defined as the expansion or shrinkage that a refractory material undergoes when it is heated up to a given temperature for a set period of time and then cooled down to room temperature (Fig. 8). The ratio between the change in length (final – initial) and the initial one is expressed as a percentage. This property is used for the design of expansion joints, and also measured for quality control purposes.

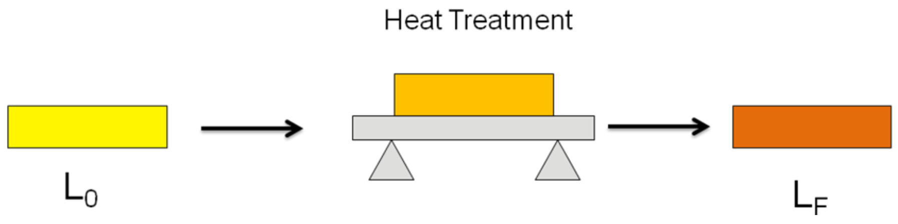


Fig. 8 - Scheme of permanent linear change test.

Depending on the type or refractory product to be tested several standards describe the test procedure:

i. EN 993-10: For dense shaped refractories.
ii. EN 1094-6: For insulating shaped refractories.
iii. EN-ISO 1927-6: For unshaped refractories

It is worthwhile to note that although unshaped refractories PLC is described in EN-ISO 1927-6, sample preparation and firing conditions are specified in EN-ISO 1927-5.

Factors affecting PLC

The investigation of the influencing factors on permanent linear change was carried out in two phases: the first one involving one laboratory and the second one four. As Fig. 9 shows many potential influencing factors can be identified. To mention just a few: sample preparation, heating schedule, apparatus involved in temperature and length measurement, etc. The purpose of the investigation was to evaluate the factors that might be fixed or modified in the current standards without involving a complete dispensable and/or counter-productive revolution of the testing method.

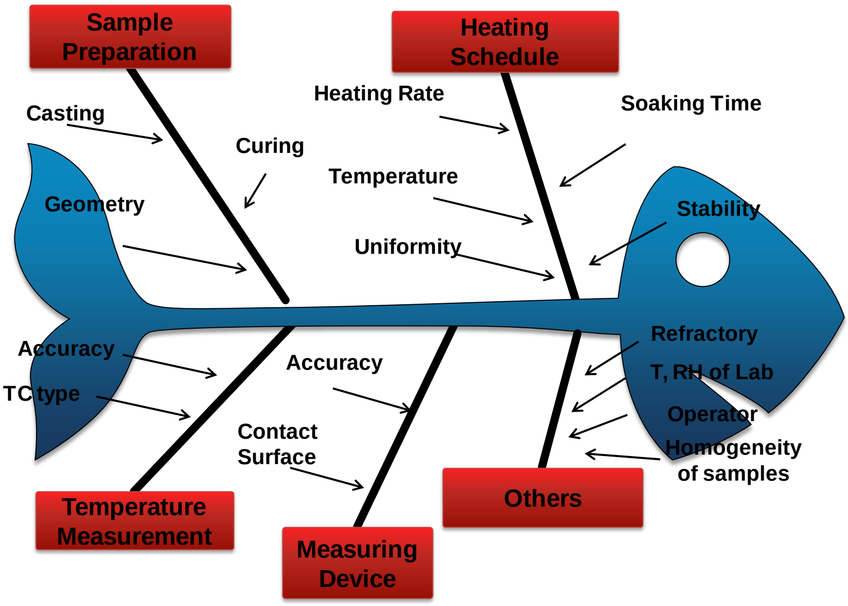


Fig. 9 - Fishbone chart showing some potential influencing factors in PLC.

Since not all factors could be extensively studied, some had to be set. The factors fixed were

i. Temperature of the PLC tests.
ii. Type of materials tested.
iii. Accuracy of length measuring devices.
iv. Maximum temperature gradient allowed during thermal treatment.
v. Minimum uniformity required in the thermal treatment.
vi. Accuracy of the temperature measurement.
vii. Sample preparation procedure.

The factors that were studied in detail were selected according to the experience of the laboratories involved in the project, their theoretical impact in the uncertainty of PLC, and those thought to be inappropriately fixed in the current standards. This included:

i. Measurement device: Dial Gauge, Height Gauge and Calipers were compared
ii. Heating rate
iii. Soaking time
iv. Geometry and direction of extraction for shaped refractory products
v. Test piece format for unshaped refractory products
vi. Cooling rate

In addition, the factor operator was investigated in order to guarantee the goodness of the results obtained.

Summary of the method specific investigations and findings

In the initial phase of the project, a full factorial design with a general linear model including up to second order interaction was set up and performed. This permitted the identification of the importance of main effects and second order interactions which were expected to be important.

Due to the fact that permanent linear change is a time consuming test, in the second phase of the project, a fractional factorial design was implemented. This approach did however not permit identification of the importance of second order interactions due to the reduced resolution of the design. Tab. 2 shows the main influencing factors identified for each Standard. Generally speaking, the heating rate was the most important factor, except for refractory castables where the format of the test specimen was found to be extremely significant.

Tab. 3 - Influencing factors identified for PLC.

EN 993-10	EN 1094-6	EN-ISO 1927-6
Heating Rate	Heating Rate	Geometry
Measuring method	Measuring method	Heating Rate
Length		Soaking Time

Once the influencing factors had been identified, an improved testing methodology was established and later on followed in an interlaboratory test involving all the partners of the project (phase 3). In this exercise dense and insulating bricks, and dense and insulating castables were included in order to obtain precision data for all these types of refractory materials. Reproducibility and Repeatability obtained for bricks are similar to those provided by other normative bodies as ASTM. The results obtained for refractory castables shown that:

i. Repeatability obtained for dense castables was lower than for dense bricks, while reproducibility of PLC was higher. This result highlights the influence of sample preparation of the castables leading to a higher dispersion in the PLC values among laboratories.
ii. For insulating castables approximately the same reproducibility than for insulating firebricks was obtained, while the repeatability was much lower. This fact is due to the high surface roughness of the insulating bricks compared with the insulating castables, resulting in more dispersion of PLC values in the former case.

The obtained precision data are expected to be included in a future revised EN PLC Standards.

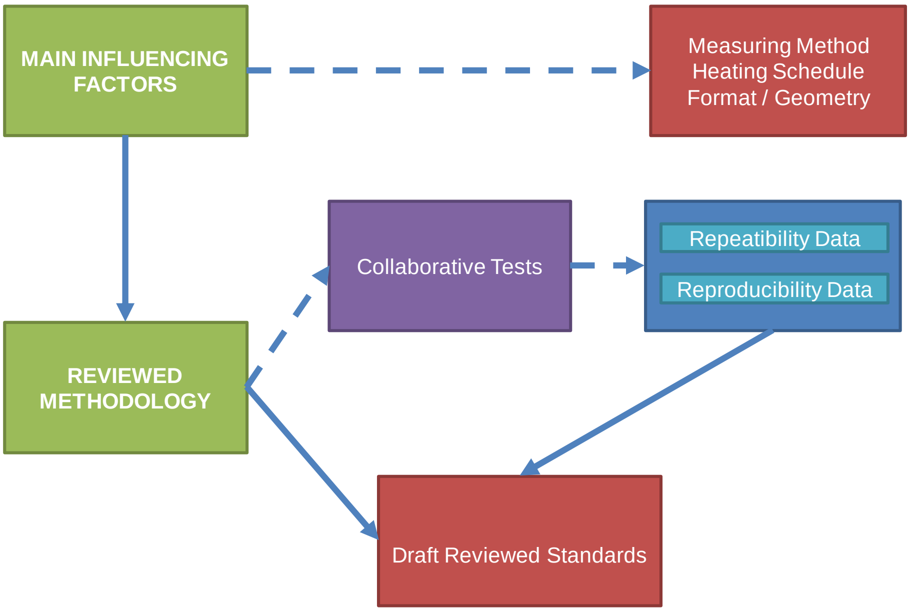


Fig. 10 - Workflow concerning PLC test.

Proposition of improvements for the EN standard & implications

The improved testing methodology has been included in a drafted of the standards. The main changes are related with the influencing factors identified:

- Heating Schedule: Depending of the type of refractory tested the variation in the heating rate could affect the permanent linear change values obtained. Therefore, a more restrictive heating schedule has been proposed
- Geometry of test specimens: Since in statistical terms the geometry of the test pieces used could affect PLC. The reviewed standard would require that a comparison between PLC values is made only if the same geometry of test specimens was used.
- Measuring devices and methods: guidelines for the measuring devices selection for EN 1094-6 have been included. Also the effect of change the reference method for dense bricks PLC is highlighted in the reviewed standards.

Another changes related with the metrological requirement on the apparatus and the statistical goodness of the results have been included:

- Metrological requirements of the apparatus: accuracy and scale division of the apparatus used in the measuring of length and temperature have been defined. For instance an accuracy of at least ±5 °C for the continuous record of the temperature has been proposed.
- Number of test specimens: Since one of the intended uses of the standards is providing PLC data for technical data sheets, an increase in the number of test specimens is required to obtain a meaningful value.

1.3.3.5. Refractoriness under Load

According to EN ISO 1893, Refractoriness under load (RuL) is a method for determining the deformation of dense and insulating refractory products, when subjected to a constant load under conditions of progressively rising temperature, by a differential method. The main characteristic of the testing procedure are strictly described in standards, which are EN ISO 1893 [2007] for shaped materials and EN ISO 1927-6 [2012] for unshaped materials. The temperature T_0 corresponding to the begin of investigation test piece subsidence under standardized RuL testing conditions is one of the most important parameters of a refractory material, since it provides access to a maximal operating temperature for the investigated material, i.e. around T_0 -200°C. Altogether, there is general agreement that the testing method contributes to the safety of high temperature installations: for instance ceramic kiln, blast furnace, boiler, casting ladle, incinerator etc.

Investigated testing parameters

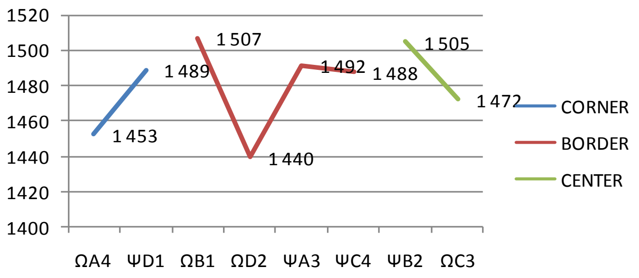
Taking into account the RuL equipment, the process conditions and the test piece preparation, a large number of parameters are potentially of interest to improve the current standard. The most relevant were investigated to quantify their effects and signal values and/or noise. For example, the dimensions of the test-piece (height, diameter) was investigated in regard to potential for saving testing material. Machining aspects as extracting and then grinding of the test-piece could be simplified for the first and removed for the second, if such a possibility was scientifically proved, which could bring more convenience in use for the testing. Finally, the testing method could gain more flexibility with the relaxation of process conditions as loading stress applied to the test-piece and position of the thermocouple (required for the measurement of temperature inside the test-piece).

Summary of the method specific investigations and findings

During the first phase of the project, fractional designs with up to eight investigated testing parameters (factors) were implemented for the RuL testing method in order to identify the most relevant factors influencing the RuL measurement results in terms of signal value and noise. Since the RuL testing method is labor and energy intensive as well as time-consuming the realization of a complete plan would have been unrealistic due to the very high number of experiment required. Fractional designs are a very good compromise of minimization of experimental cost and maximization of data. Three types of materials have been considered as a representative selection of the existing range of refractory materials: a dense brick, an insulating brick and a dense castable. The campaign represented circa 80 experiments in total. As expected, the loading was found to significantly affect RuL data, whatever the considered characteristic temperature (T_0, $T_{0,5}$, T_1 and T_2, corresponding to specific degree of subsidence of the test piece, respectively 0,5 %, 1 % and 2 %). Height had also a quite global effect, whatever the materials involved. The campaign demonstrated that the grinding step should not be a priori overlooked. Finally, the analysis of the position of the specimen axis toward the shaping direction indicated interesting perspectives.

The second phase of the project aimed at finding more optimized values for the parameters with presenting potential for improvement selected from the first phase of the project (see section 1.3.2.2 Phase 2: Appraisal of the influencing factors). As regards to the RuL specific investigations, four different laboratories were involved in three factorial designs (each with 4 factors with 2 levels). 72 RuL measurements were accordingly performed. For the analysis of effects of **high alumina dense bricks**: significant effects differ from one lab to another, which raised relevant questions about reproducibility of the testing method. The [load] has a significant (negative) effect whatever the statistical treatment used, which means that the more load is applied to the specimen, the lower T_0 temperature is obtained. Depending on the treatment of results, [use of grinding] has a significant (positive) effect if an individual treatment is considered (laboratory per laboratory), while [none-use of platinum sheet] has a significant (positive) effect if we consider a global treatment (all results pooled together). The second part consisted in analyzing the repeatability / reproducibility of the results. [location of thermocouple] affected the quality of RuL results both in terms of repeatability and reproducibility, which was also showed by the noise effect analysis. In any case, this represents a surprising result, which needs to be corroborated by further experiments. [use of grinding] and [non-centered thermocouple] lead globally to better values of dispersion, while [use of grinding] and [non-use of platinum sheet] improve repeatability (noise effect analysis).

The third phase of investigation was an extensive program of collaborative tests that involved 8 different laboratories in case of the RuL testing method. In addition to the refractory products already considered during the phase 1 & 2, an insulating unshaped refractory products was added to the investigation. Each partner involved in the collaborative tests performed 28 RuL measurements. In order to investigate a potential effect of the position of the test-piece in the initial **dense brick**, test pieces have been drilled either from the center, or from the border or from the corner of the initial brick. The set of corresponding results does not present any correlation between $T_{0.5}$ values and position of the test-piece within the bricks.



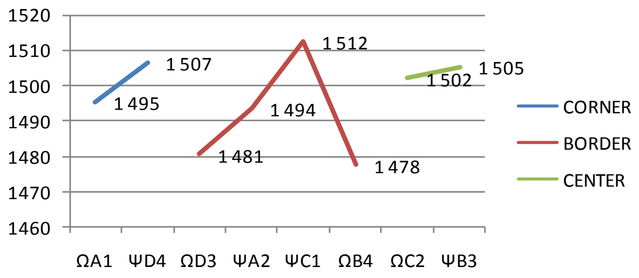


Fig. 11 – Graphical representation of the RuL testing results from two different laboratory in order to identify correlation between position of test-piece and $T_{0.5}$ values of dense bricks

The coefficient of thermal expansion α was calculated from the RuL curves in order to have some elastic parameters to study in addition to the elasto-visco-plastic ones (i.e. RuL characteristically temperatures). The values for the thermal expansion α correspond to the slope of the RuL curves, they can be roughly estimated in dividing the value of maximum linear expansion by the temperature T_0 (Fig. 12). As already observed with RuL characteristically temperatures, no clear correlation between α values and position of test-piece (centre, corner) was observed.

For **insulating bricks**, the effect analysis of the position of the test-piece in the initial brick indicates clearly that the differential corner/centre is negligible compared with the heterogeneity between bricks. Actually, this adds more flexibility in the step of test piece extraction.

The last but not the least objective of the phase 3 was to produce sufficient data in order to have relevant statistical treatments for informational purpose to the potential user. The precision data are now included in the drafts of the revised standards for the RuL testing methods.

Proposition of improvements for the EN standard & implications

Clarifying the purpose of the test: The temperature T_0 extracted from RuL test is one of the most important parameters of a refractory material, as it provides access to a maximal operating temperature, which could be around T0 -200°C. The conditions here prescribed in the standard are not intended to strictly represent the industrial reality and the user should be aware of the significant impact of the atmosphere to the RuL behavior of the material tested.

Facilitate the use of the standard: Instead of recommending a RuL behavior for some part of the equipment as discs, column or tubes, the mention of a suited grade of the related parts is preferred.

Reducing the interpretation and potential causes of discrepancies between users: An additional note informs the user of the potential effect of a platinum sheet (see section 5. Apparatus / 5.1 Loading device / 5.1.4 Two discs). More precisely, the temperatures obtained according such a mounting are likely to be significantly lower than the ones obtained without foil. Indeed, the current text of the standard recommends the use of a platinum foil between the discs and the test piece in the case of suspicion of chemical reaction between them. Especially for dense materials, the use of a platinum foil may have an impact on testing results.

Checking the elastic part of RuL curve via the estimation of the coefficient α of thermal expansion of the material (Fig. 12) tested appeared to be a pertinent step prior to the analysis of RuL values.

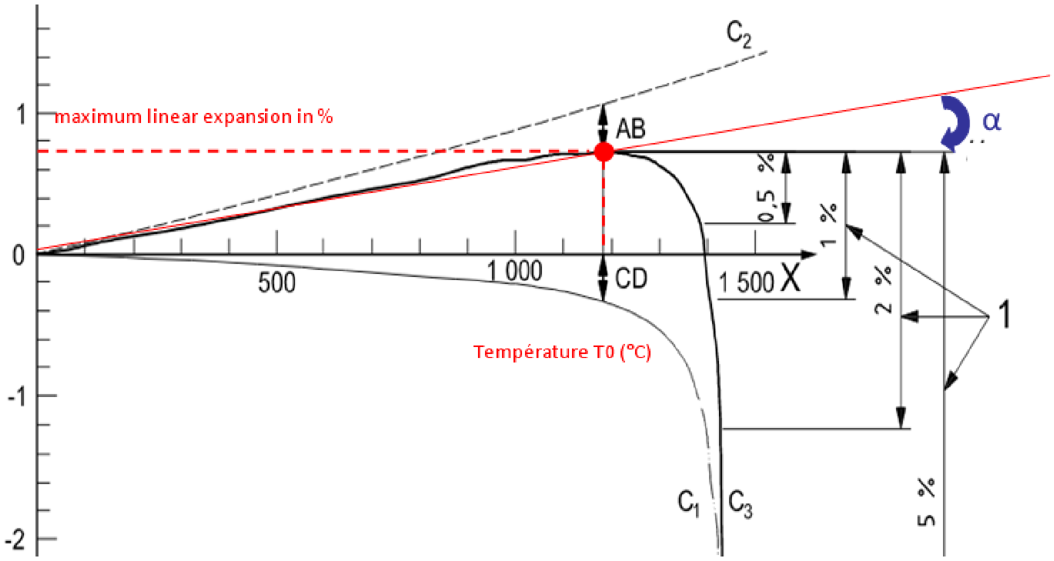


Fig. 12 – Evaluation of coefficient alpha of thermal expansion (X-axis: temperature in °C – Y-axis: linear expansion in %)

Calibration of the equipment: check if the raw curve is confused with zero line in case of measure of high grade alumina test-piece (which should be the same grade of the alumina from the tubes). If there is a deviation from the zero-line, it is highly recommended to replace the tubes with new ones of appropriate grade, as ageing of tubes is a common cause of such a deviation. Notice that a deviation may also have other causes as ageing of thermocouple, malfunction of transducer or misalignment of tubes/columns/test-piece system.

Correction curve: use strictly the expansion curve relative to the grade of which the alumina tube is made, instead of generic data.

Incorporating the generated precision data: This part is included in the last section of the text of the standard under the form of a chart presenting statistical data from the collaborative tests performed within ReStaR project. A foreword specifies the different aspects of the collaborative tests purpose and framework in order to inform the user of the strict conditions of data generation. Especially for RuL measurements, every tests are expensive and time-consuming, very few laboratories can allow themselves to generate enough experimental data to establish sound repeatability data even for one material, not to mention the need to involve independent laboratories for reproducibility data. The purpose of such an additional section to the standard is to propose, for potential user of the testing method, a statistical framework which may help him to have an order of magnitude of the acceptable dispersion of his results concerning a very similar material.

1.4. Potential and the main dissemination activities and exploitation of results

1.4.1. potential impact

Drafts of the reviewed testing Standards

The results from the RTD activities, i.e. the investigations of the influencing factors for the investigated EN testing methods, highlighted improvement or simplification potential for the corresponding EN testing standards. The proposed drafts of the EN testing standards includes: clarification of the purpose of the test, guidance notes to support the user in some choices and facilitate the use of the standard, as well as reducing the interpretation and potential causes of discrepancies between users, and finally incorporate the generated precision data. Robustly and pragmatically revised EN testing standards will gained in acceptance in Europe and worldwide.

In recent years, the European refractory industry has experienced considerable pressure from overseas competitors, especially Brazil and China. The European refractory production is still characterised by a superior level of quality. It is important that EN testing standards are able to reflect this level of product quality and product performance. Especially European start-up SMEs with limited international reputation would benefit of recognized and reliable EN testing standards to promote the quality of their product in Europe.

The price of refractories themselves only represents a small part of the materials production cost (generally some %) in the industry where they are used. However, the cost of production stoppages is usually not negligible and can be very expensive depending on the extent of the damage and the duration. Unexpected refractory failures lead to plant breakdowns with expensive production stop, loss of time, production, materials and labour. Once again reliable testing standard are needed to properly assess the quality of a refractory products and avoid unnecessary discussions between refractory producers and user about the precision of the measurement results according to EN testing standards.

The human safety is also an important aspect to guarantee for the workers of the production site, a reliable characterization of the refractory quality may reduce the likelihood of tragic accidents.

In addition, standards tend to increase competition and allow lower output and sales cost, benefiting economies as a whole. A recent study issued by one of the major national standards bodies has asserted that in Europe standardisation adds approximately 1 % to the value of the product [1]. However, ageing standards lose recognition and acceptance. Some end-users develop their own testing methods, which are declared as a reference although they have not been validated. Hereby measurement results of doubtful relevance and significance are then defined as a condition of supply. Depending on the scope of supply of the rejected product, cost totalling hundreds of thousands of Euros may be incurred.

The CEN estimates that the benefit of the development of European standards and keeping them at the state-of-the-art is 10 to 20 times higher than the involved cost [1].

As from more accurate reviewed testing, SMEs manufacturers with limited experience in material characterisation will be enabled to provide standards-compliant property values of their products with minor deviation and inaccuracy. Therefore they will be able to better guarantee the quality of their production.

Besides, many refractory laboratories, which often belong to SME manufacturers or are themselves independent SMEs, maintain obsolete testing systems to meet test specifications requested by each customer. The costs for quality assuring measures are thus consequently increased. World-wide recognised testing standards would minimise such costs and increase the competitiveness of refractory producers SMEs.

Methodology to review testing standards

Besides the establishment of Drafts of the reviewed testing Standards, the methodology followed during the ReStaR to review testing standards was documented in a specific report. This document summarizes the steps implemented as well as the accumulated experience, compiling lessons learned and good practices, to build on. It is a supporting tool for consortia aiming at reviewing testing standards, containing guidelines for similar standard reviewing activities.

The following audiences may take benefice from the report to respectively fulfil the following objectives:

- Standardisation bodies: to provide national or international standardization bodies or more especially their technical committees and working groups, with recommendations for the revising and amending of existing testing standards or for setting new ones;
- Consortia aiming at reviewing testing standards: to pass on experience gained in the ReStaR project and provide an overview of activities, tools, good practices and advices for future testing standard reviewing works.

1.4.2. main dissemination activities

Various dissemination channels and media were considered and aimed to obtain maximum impact from promotion of the ReStaR project. The channels are selected according to the intended audience. For promotion of the project and its activities, different media were used, adapted for different target groups (Tab. 4).

Tab. 4 - Types of activities used to disseminate ReStaR results according to different target groups

Target group / Tool	EC	Authority	Industry	Research sector	General public	Standard. Bodies
Website	x	x	x	x	x	x
Deliverables - restricted	x					
Deliverables - public	x	x	x	x	x	x
Technical and scientific publication			x	x		x
Dedicated workshop, Symposia, etc	x	x	x	x		x
Trade shows		x	x			
Technical fairs		x	x			
Congress	x	x	x	x		x
Stakeholder		x	x	x		x

forums		
Newsletters	x	x

Logo

The project logo (Fig. 13) defines the project visual identity, creates an easily recognisable “image” and helps to improve the visibility. It was used prominently in all dissemination tools and printed materials.

Fig. 13 – ReStaR logo

E-Media

Project website: The ReStaR project website is a substantial tool for the dissemination process, addressing a large audience. The general public can find information about the refractory sector and become aware of key role of refractory products for our modern society: “the world's most important but least known products” (Charles E. Semler). Furthermore, the ReStaR project website provides specific details on the project aims and objectives. The refractory industry stakeholders can learn about the issues and progresses of the research project. Finally, the actors of the research sector will find a list and information on the scientific and technical publications worked out in the framework of the project.

The website is based on a simple and clear structure, to promote an effective and pleasant navigation.

Partner’s websites: Most partners added a web link to the project website and/or published non-confidential contents on their own company website.

Newsletters: The European SME AG, CU / PRE, and some ReStaR partners used their corporate newsletters to disseminate update on the project progress. Newsletters are simple but efficient vector of information. PRE newsletters are sent to all PRE members 5 times per year. Especially SMEs can be directly targeted in this way. National SME AGs member of PRE were also encouraged to relay and relayed the information of interest in their own national language(s). The Cerame-Unie newsletter is distributed twice a year to a large audience in the ceramic industry and European policy makers.

Events

Presentations at relevant workshops, trade shows, technical fairs and other conferences were an important promotional channel for publishing technical and scientific research results. The most significant events are listed below:

- RTD performers presented selected results during the following international conference: 57th International Colloquium on Refractories in Aachen (Germany), XVI International Scientific Conference Refractory materials in Wisla (Poland), 14th Biennial Worldwide Congress UNITECR 2015 in Vienna (Austria).

- Presentation of the project progress at the PRE Technical Committee meetings, PRE Congresses and Ceramic Days 2014.
- The CU / PRE, in collaboration with ECREF, managed to organize a special ReStaR session during the International Technical Conference on Refractories (UNITECR 2015) in Vienna on the 18th of September 2015. UNITECR has established itself as the most important international panel for scientific and technical matters in the refractory world. In addition to direct participants from the refractory industry, raw material suppliers, consumers and scientists also have a great opportunity to participate in discussions, exchange information and even debate on the best path for the future of our industry.

Publications

Opportunities were sought to publish articles in relevant scientific and technical journals:

- Two "stand-alone" articles were published in the journal Refractories WORLDFORUM. Refractories WORLDFORUM is a technical and scientific journal that strive for building a bridge between industry and science.
- A bundle of articles was issued in the technical and scientific journal Refractories WORLDFORUM by the end of the project.
- A Technical article was issued in the scientific journal Materiały Ceramiczne.

Other dissemination channels

For the benefit of SMEs, specific measures were implemented. The ReStaR project partners sought opportunities to visit SMEs, on one hand to better understand their needs, and on the other hand to directly pass on project results and experience. Especially, technicians and engineers from SMEs working on almost a daily basis with the testing method were targeted.

1.4.3. exploitation of results

Standard adoption procedure

The revision of the relevant EN testing standards was officially initiated at the CEN / TC 187 Technical Committee for refractory product meeting on the 3rd March 2015. Drafts of the revised relevant EN testing standards were provided the CEN / TC 187 for the next technical meeting planned for the beginning of 2016.

Once a draft of a European Standard is prepared, it is released for public comment, a process known in CEN as the 'CEN Enquiry'. During the public commenting stage, everyone who has an interest (e.g. manufacturers, public authorities, consumers, etc.) may comment on the draft.

Taking into account the comments resulting from the CEN Enquiry, a final version is drafted which is then submitted to the 30 CEN Members for a weighted Formal Vote.

The submission process may start by the end of the project and extend beyond the end of the project (Fig. 14). However, this is the main priority of the project exploitation activities.

As soon as the reviewed testing Standard drafts acquire the full status of EN Standards, the CU / PRE will promote the adoption of the sound reviewed EN Standards as ISO Standards. The CU / PRE is the only organization with a *Liaison A* with the ISO technical committee for refractory products (ISO/TC 33). With regard to the ISO's structure, organisations with a *Liaison A* make an effective contribution to the work of the technical committee for questions dealt with by this technical committee.

In this task, the CU / PRE will be supported by the project members which also participate to the ISO/TC 33.

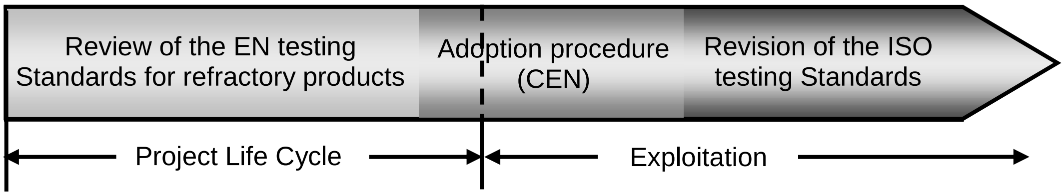


Fig. 14 – Standardisation work

1.5. Project public website

The project public website is accessible through: www.restar.eu

References

[1] The Economic Benefits of Standardization, An update of the study carried out by DIN in 2000, published by DIN (German Institute for Standardisation) (2011)

[2] A. Majdic, L. Hagemann, H. Lichomski, "Einfluss der gute der probekörperdruckflächen und der Druckplattenrauheit auf Mittlwer und Streubreite der Klatdruckfestigkeit Steine", Tonind. Zeit, 97, 1973, 9, 237-243

4.1 Final publishable summary report

This section must be of suitable quality to enable direct publication by the Commission and should preferably not exceed 40 pages. This report should address a wide audience, including the general public.

The publishable summary has to include **5 distinct parts** described below:

- An executive summary (not exceeding 1 page).
- A summary description of project context and objectives (not exceeding 4 pages).
- A description of the main S&T results/foregrounds (not exceeding 25 pages),
- The potential impact (including the socio-economic impact and the wider societal implications of the project so far) and the main dissemination activities and exploitation of results (not exceeding 10 pages).
- The address of the project public website, if applicable as well as relevant contact details.

Furthermore, project logo, diagrams or photographs illustrating and promoting the work of the project (including videos, etc…), as well as the list of all beneficiaries with the corresponding contact names can be submitted without any restriction.

4.2 Use and dissemination of foreground

A plan for use and dissemination of foreground (including socio-economic impact and target groups for the results of the research) shall be established at the end of the project. It should, where appropriate, be an update of the initial plan in Annex I for use and dissemination of foreground and be consistent with the report on societal implications on the use and dissemination of foreground (section 4.3 – H).

The plan should consist of:

- Section A

  This section should describe the dissemination measures, including any scientific publications relating to foreground. **Its content will be made available in the public domain** thus demonstrating the added-value and positive impact of the project on the European Union.

- Section B

  This section should specify the exploitable foreground and provide the plans for exploitation. All these data can be public or confidential; the report must clearly mark non-publishable (confidential) parts that will be treated as such by the Commission. Information under Section B that is not marked as confidential **will be made available in the public domain** thus demonstrating the added-value and positive impact of the project on the European Union.

2. Use and dissemination of foreground

2.1. Section A (public)

This section includes two templates

- Template A1: List of all scientific (peer reviewed) publications relating to the foreground of the project.
- Template A2: List of all dissemination activities (publications, conferences, workshops, web sites/applications, press releases, flyers, articles published in the popular press, videos, media briefings, presentations, exhibitions, thesis, interviews, films, TV clips, posters).

These tables are cumulative, which means that they should always show all publications and activities from the beginning until after the end of the project. Updates are possible at any time.

TEMPLATE A1: LIST OF SCIENTIFIC (PEER REVIEWED) PUBLICATIONS, STARTING WITH THE MOST IMPORTANT ONES										
NO.	Title	Main author	Title of the periodical or the series	Number, date or frequency	Publisher	Place of publication	Year of publication	Relevant pages	Permanent identifiers[1] (if available)	Is/Will open access[2] provided to this publication?
1	*Concerted Effort in the European Refractory Sector to Consolidate and*	*E. Brochen*	*Refractories WORLDFORUM*	*7 (2015) [3]*	*Göller Verlag GmbH*	*Germany*	*2015*	*85-86*		no

[1] A permanent identifier should be a persistent link to the published version full text if open access or abstract if article is pay per view) or to the final manuscript accepted for publication (link to article in repository).
[2] Open Access is defined as free of charge access for anyone via Internet. Please answer "yes" if the open access to the publication is already established and also if the embargo period for open access is not yet over but you intend to establish open access afterwards.

	Make EN Testing Standards Future-Proof									
2	*Investigation of the Parameters Influencing the Refractoriness under Load (RuL) Testing Results for Refractory Materials*	*A. Stuppfler*	*Refractories WORLDFORUM*	*7 (2015) [3]*	*Göller Verlag GmbH*	*Germany*	*2015*	*87-93*		no
3	*Investigation of the Factors Influencing the Bulk Density and Open Porosity Testing Results for Refractory Materials*	*E. Dahlem*	*Refractories WORLDFORUM*	*7 (2015) [3]*	*Göller Verlag GmbH*	*Germany*	*2015*	*95-104*		no
4	*Investigation of the Testing Parameters Influencing the Cold Crushing Strength Testing Results of Refractory Materials*	*J. Czechowski*	*Refractories WORLDFORUM*	*7 (2015) [3]*	*Göller Verlag GmbH*	*Germany*	*2015*	*105-112*		no
5	*Investigation of Testing Parameters Influencing the Permanent Linear Change Testing Results of Dense Refractory Bricks*	*J. R. Campello-García*	*Refractories WORLDFORUM*	*7 (2015) [3]*	*Göller Verlag GmbH*	*Germany*	*2015*	*113-118*		no
6	*Investigation of the Main Factors Susceptible to Influence the Modulus of Rupture Testing Results of Refractory Materials*	*J.P. Erauw*	*Refractories WORLDFORUM*	*7 (2015) [4]*	*Göller Verlag GmbH*	*Germany*	*2015*	*95-103*		no
7	*The importance of test piece preparation of unshaped refractory products for the characterization of physical properties according to en iso standards*	*V. Halapa*	*Refractories WORLDFORUM*	*7 (2015) [1]*	*Göller Verlag GmbH*	*Germany*	*2015*	*73-81*		no
8	*An analysis of factors influencing the determination of refractory materials' compression strength*	*A. Gerle*	*Materiały Ceramiczne*	*?*	*?*	*Poland*	*2015*	*?*		yes/no
9	*ReStaR – Collaborative R&I in*	*C. Dannert*	*Refractories*	*5 (2013) [4]*	*Göller*	*Germany*	*2013*	*41-42*		no

	the European refractory sector to review and improve testing standards		*WORLDFORUM*		*Verlag GmbH*					

TEMPLATE A2: LIST OF DISSEMINATION ACTIVITIES

NO.	Type of activities[3]	Main leader	Title	Date/Period	Place	Type of audience[4]	Size of audience	Countries addressed
1	*publication*	*A. Volckaert*	*PRE newsletter - issue 22*	*15th October 2013*	-	*Industry*	*> 130 European companies from the refractory industry*	Europe
2	*web*	*E. Dahlem*	www.restar.eu	*December 2013*	-	*Medias*		Europe
3	*publication*	*A. Volckaert*	*Cerame-Unie newsletter issue 2*	*January 2014*	-	*Industry*	*> 2000 European companies from the ceramic industry*	Europe
4	*Other*	*A. Volckaert*	*Cerame-Unie Activity report 2013*	*April 2014*	-	*Industry*	*> 2000 European companies from the ceramic industry*	Europe
5	*presentation*	*A. Volckaert*	*PRE Technical Committee meeting*	*10th March 2014*	*Brussel, Belgium*	*Industry*	*?*	Europe
6	*presentation*	*A. Volckaert*	*PRE Congress*	*4th-6th June 2014*	*Porterož, Slovenia*	*Industry*	*?*	Europe

[3] A drop down list allows choosing the dissemination activity: publications, conferences, workshops, web, press releases, flyers, articles published in the popular press, videos, media briefings, presentations, exhibitions, thesis, interviews, films, TV clips, posters, Other.

[4] A drop down list allows choosing the type of public: Scientific Community (higher education, Research), Industry, Civil Society, Policy makers, Medias, Other ('multiple choices' is possible).

7	*Publication*	*G. Urbanek*	*RHI Bulletin>1>2014*	*July 2014*	-	*Industry*		World
8	*publication*	*A. Volckaert*	*Cerame-Unie newsletter issue 3*	*July 2014*	-	*Industry*	*> 2000 European companies from the ceramic industry*	Europe
9	*presentation*	*E. Brochen*	*German Standards committee (DIN) for refractory product meeting*	*17th September 2014*	*Höhr-Grenzhausen*	*Industry*	*9 representatives from the German refractory industry*	Germany
10	*publication*	*A. Gulasci*	*Standardization and Industrial Leadership*	*September 2014*	-	*Industry, Policy makers*	-	Europe
11	*conference*	*E. Dahlem*	*the 57th International Colloquium on Refractories*	*25th September 2014*	*Aachen, Germany*	*Industry,*		World
12	*Presentation*	*A. Volckaert*	*Ceramic Days 2014*	*3-4 December 2014*	*Brussels, Belgium*	*Industry, Policy makers*	*> 150 representatives from the European ceramic industry*	Europe
13	*publication*	*A. Volckaert*	*PRE newsletter - issue 28*	*15th December 2014*	-	*Industry*	*> 130 European companies from the refractory industry*	Europe
14	*presentation*	*A. Volckaert*	*PRE Technical Committee meeting*	*4th March 2015*	*Industry*	*Industry*	*?*	Europe
15	*publication*	*A. Volckaert*	*Cerame-Unie newsletter issue 4*	*January 2015*	-	*Industry*	*> 2000 European companies from the ceramic industry*	Europe
16	*presentation*	*E. Dahlem*	*CEN / TC 187 Technical Committee for*	*3rd March 2015*	*Brussels, Belgium*	*Industry*	*14 representatives from the*	Europe

			refractory product meeting				European refractory industry	
17	presentation	J. Podwórny	CEN / TC 187 Technical Committee for refractory product meeting	3rd March 2015	Brussels, Belgium	Industry	14 representatives from the European refractory industry	Europe
18	presentation	J.P. Erauw	CEN / TC 187 Technical Committee for refractory product meeting	3rd March 2015	Brussels, Belgium	Industry	14 representatives from the European refractory industry	Europe
19	presentation	J. R. Campello-García	CEN / TC 187 Technical Committee for refractory product meeting	3rd March 2015	Brussels, Belgium	Industry	14 representatives from the European refractory industry	Europe
20	presentation	A. Stuppfler	CEN / TC 187 Technical Committee for refractory product meeting	3rd March 2015	Brussels, Belgium	Industry	14 representatives from the European refractory industry	Europe
21	Other	A. Volckaert	Cerame-Unie Activity report 2014	May 2015	-	Industry	> 2000 European companies from the ceramic industry	Europe
22	other	A. Volckaert	PRE Annual report 2014	May 2015	-	Industry	> 130 European companies from the refractory industry	Europe
23	conference	E. Dahlem	XVI International Scientific Conference Refractory materials	20-22 May 2015	Wisla, poland	Industry	?	Europe

24	*conference*	*A. Gerle*	*XVI International Scientific Conference Refractory materials*	*20-22 May 2015*	*Wisla, poland*	*Industry*	*?*	Europe
25	*conference*	*A. Stuppfler*	*XVI International Scientific Conference Refractory materials*	*20-22 May 2015*	*Wisla, poland*	*Industry*	*?*	Europe
26	*presentation*	*A. Volckaert*	*PRE Congress*	*27-29 May 2015*	*Istanbul, Turkey*	*Industry*	*?*	Europe
27	*publication*	*A. Volckaert*	*Cerame-Unie newsletter issue 5*	*July 2015*	*-*	*Industry*	*> 2000 European companies from the ceramic industry*	Europe
28	*presentation*	*V. Halapa*	*14th Biennial Worldwide Congress UNITECR 2015 in Vienna*	*15-18 September 2015*	*Vienna, Austria*	*Industry*	*> 900 representatives from the refractory industry*	World
29	*presentation*	*E. Brochen*	*14th Biennial Worldwide Congress UNITECR 2015 in Vienna*	*15-18 September 2015*	*Vienna, Austria*	*Industry*	*> 900 representatives from the refractory industry*	World
30	*presentation*	*E. Dahlem*	*14th Biennial Worldwide Congress UNITECR 2015 in Vienna*	*15-18 September 2015*	*Vienna, Austria*	*Industry*	*> 900 representatives from the refractory industry*	World
31	*presentation*	*J. Czechowski*	*14th Biennial Worldwide Congress UNITECR 2015 in Vienna*	*15-18 September 2015*	*Vienna, Austria*	*Industry*	*> 900 representatives from the refractory industry*	World
32	*presentation*	*J. R. Campello-García*	*14th Biennial Worldwide Congress UNITECR 2015 in Vienna*	*15-18 September 2015*	*Vienna, Austria*	*Industry*	*> 900 representatives from the refractory industry*	World

33	*presentation*	*J.P. Erauw*	*14th Biennial Worldwide Congress UNITECR 2015 in Vienna*	*15-18 September 2015*	*Vienna, Austria*	*Industry*	*> 900 representatives from the refractory industry*	World
34	*presentation*	*A. Stuppfler*	*14th Biennial Worldwide Congress UNITECR 2015 in Vienna*	*15-18 September 2015*	*Vienna, Austria*	*Industry*	*> 900 representatives from the refractory industry*	World
35	*presentation*	*J. Czechowski*	*SPMO (Association of Refractory Materials Manufacturers in Poland) members meeting*	*22 October 2015*	*?, Poland*	*Industry*	*?*	Poland

2.2. Section B (Confidential[5] or public: confidential information to be marked clearly)

2.2.1. Part B1

The applications for patents, trademarks, registered designs, etc. shall be listed according to the template B1 provided hereafter.

The list should, specify at least one unique identifier e.g. European Patent application reference. For patent applications, only if applicable, contributions to standards should be specified. This table is cumulative, which means that it should always show all applications from the beginning until after the end of the project.

TEMPLATE B1: LIST OF APPLICATIONS FOR PATENTS, TRADEMARKS, REGISTERED DESIGNS, ETC.					
Type of IP Rights[6]:	Confidential Click on YES/NO	Foreseen embargo date dd/mm/yyyy	Application reference(s) (e.g. EP123456)	Subject or title of application	Applicant (s) (as on the application)

[5] Note to be confused with the "EU CONFIDENTIAL" classification for some security research projects.

[6] A drop down list allows choosing the type of IP rights: Patents, Trademarks, Registered designs, Utility models, Others.

2.2.2. Part B2

Please complete the table hereafter:

Type of Exploitable Foreground[7]	Description of exploitable foreground	Confidential Click on YES/NO	Foreseen embargo date dd/mm/yyyy	Exploitable product(s) or measure(s)	Sector(s) of application[8]	Timetable, commercial or any other use	Patents or other IPR exploitation (licences)	Owner & Other Beneficiary(s) involved
	Ex: New superconductive Nb-Ti alloy			*MRI equipment*	*1. Medical 2. Industrial inspection*	*2008 2010*	*A materials patent is planned for 2006*	*Beneficiary X (owner) Beneficiary Y, Beneficiary Z, Poss. licensing to equipment manuf. ABC*

In addition to the table, please provide a text to explain the exploitable foreground, in particular:

- Its purpose
- How the foreground might be exploited, when and by whom
- IPR exploitable measures taken or intended
- Further research necessary, if any
- Potential/expected impact (quantify where possible)

[19] A drop down list allows choosing the type of foreground: General advancement of knowledge, Commercial exploitation of R&D results, Exploitation of R&D results via standards, exploitation of results through EU policies, exploitation of results through (social) innovation.
[8] A drop down list allows choosing the type sector (NACE nomenclature) : http://ec.europa.eu/competition/mergers/cases/index/nace_all.html

4.3 Report on societal implications

Replies to the following questions will assist the Commission to obtain statistics and indicators on societal and socio-economic issues addressed by projects. The questions are arranged in a number of key themes. As well as producing certain statistics, the replies will also help identify those projects that have shown a real engagement with wider societal issues, and thereby identify interesting approaches to these issues and best practices. The replies for individual projects will not be made public.

A General Information *(completed automatically when **Grant Agreement number** is entered.*	
Grant Agreement Number:	
Title of Project:	
Name and Title of Coordinator:	

B Ethics	
1. Did your project undergo an Ethics Review (and/or Screening)? • If Yes: have you described the progress of compliance with the relevant Ethics Review/Screening Requirements in the frame of the periodic/final project reports? Special Reminder: the progress of compliance with the Ethics Review/Screening Requirements should be described in the Period/Final Project Reports under the Section 3.2.2 *'Work Progress and Achievements'*	***0Yes 0No***
2. Please indicate whether your project involved any of the following issues (tick box) :	***YES***
RESEARCH ON HUMANS	
• Did the project involve children?	
• Did the project involve patients?	
• Did the project involve persons not able to give consent?	
• Did the project involve adult healthy volunteers?	
• Did the project involve Human genetic material?	
• Did the project involve Human biological samples?	
• Did the project involve Human data collection?	
RESEARCH ON HUMAN EMBRYO/FOETUS	
• Did the project involve Human Embryos?	
• Did the project involve Human Foetal Tissue / Cells?	
• Did the project involve Human Embryonic Stem Cells (hESCs)?	
• Did the project on human Embryonic Stem Cells involve cells in culture?	
• Did the project on human Embryonic Stem Cells involve the derivation of cells from Embryos?	
PRIVACY	
• Did the project involve processing of genetic information or personal data (eg. health, sexual lifestyle, ethnicity, political opinion, religious or philosophical conviction)?	
• Did the project involve tracking the location or observation of people?	
RESEARCH ON ANIMALS	
• Did the project involve research on animals?	
• Were those animals transgenic small laboratory animals?	
• Were those animals transgenic farm animals?	

• Were those animals cloned farm animals?	
• Were those animals non-human primates?	
RESEARCH INVOLVING DEVELOPING COUNTRIES	
• Did the project involve the use of local resources (genetic, animal, plant etc)?	
• Was the project of benefit to local community (capacity building, access to healthcare, education etc)?	
DUAL USE	
• Research having direct military use	0 Yes 0 No
• Research having the potential for terrorist abuse	

C Workforce Statistics

3. Workforce statistics for the project: Please indicate in the table below the number of people who worked on the project (on a headcount basis).

Type of Position	Number of Women	Number of Men
Scientific Coordinator		
Work package leaders		
Experienced researchers (i.e. PhD holders)		
PhD Students		
Other		

4. How many additional researchers (in companies and universities) were recruited specifically for this project?	
Of which, indicate the number of men:	

D Gender Aspects

5. Did you carry out specific Gender Equality Actions under the project?	○ ○	Yes No

6. Which of the following actions did you carry out and how effective were they?

	Not at all effective				Very effective
❑ Design and implement an equal opportunity policy	○	○	○	○	○
❑ Set targets to achieve a gender balance in the workforce	○	○	○	○	○
❑ Organise conferences and workshops on gender	○	○	○	○	○
❑ Actions to improve work-life balance	○	○	○	○	○
○ Other:					

7. Was there a gender dimension associated with the research content – i.e. wherever people were the focus of the research as, for example, consumers, users, patients or in trials, was the issue of gender considered and addressed?

- ○ Yes- please specify
- ○ No

E Synergies with Science Education

8. Did your project involve working with students and/or school pupils (e.g. open days, participation in science festivals and events, prizes/competitions or joint projects)?

- ○ Yes- please specify
- ○ No

9. Did the project generate any science education material (e.g. kits, websites, explanatory booklets, DVDs)?

- ○ Yes- please specify
- ○ No

F Interdisciplinarity

10. Which disciplines (see list below) are involved in your project?

- ○ Main discipline[9]:
- ○ Associated discipline[9]: ○ Associated discipline[9]:

G Engaging with Civil society and policy makers

11a Did your project engage with societal actors beyond the research community? *(if 'No', go to Question 14)*	○ ○	Yes No

11b If yes, did you engage with citizens (citizens' panels / juries) or organised civil society (NGOs, patients' groups etc.)?

- ○ No
- ○ Yes- in determining what research should be performed
- ○ Yes - in implementing the research
- ○ Yes, in communicating /disseminating / using the results of the project

[9] Insert number from list below (Frascati Manual).

11c In doing so, did your project involve actors whose role is mainly to organise the dialogue with citizens and organised civil society (e.g. professional mediator; communication company, science museums)?	○ ○	Yes No

12. Did you engage with government / public bodies or policy makers (including international organisations)

- ○ No
- ○ Yes- in framing the research agenda
- ○ Yes - in implementing the research agenda
- ○ Yes, in communicating /disseminating / using the results of the project

13a Will the project generate outputs (expertise or scientific advice) which could be used by policy makers?

- ○ Yes – as a **primary** objective (please indicate areas below- multiple answers possible)
- ○ Yes – as a **secondary** objective (please indicate areas below - multiple answer possible)
- ○ No

13b If Yes, in which fields?

Agriculture Audiovisual and Media Budget Competition Consumers Culture Customs Development Economic and Monetary Affairs Education, Training, Youth Employment and Social Affairs		Energy Enlargement Enterprise Environment External Relations External Trade Fisheries and Maritime Affairs Food Safety Foreign and Security Policy Fraud Humanitarian aid		Human rights Information Society Institutional affairs Internal Market Justice, freedom and security Public Health Regional Policy Research and Innovation Space Taxation Transport	

13c If Yes, at which level? ○ Local / regional levels ○ National level ○ European level ○ International level	

H Use and dissemination

14. How many Articles were published/accepted for publication in peer-reviewed journals?	
To how many of these is open access[10] provided?	
How many of these are published in open access journals?	
How many of these are published in open repositories?	
To how many of these is open access not provided?	
Please check all applicable reasons for not providing open access:	
❑ publisher's licensing agreement would not permit publishing in a repository ❑ no suitable repository available ❑ no suitable open access journal available ❑ no funds available to publish in an open access journal ❑ lack of time and resources ❑ lack of information on open access ❑ other[11]: ……………	

15. How many new patent applications ('priority filings') have been made? *("Technologically unique": multiple applications for the same invention in different jurisdictions should be counted as just one application of grant).*		
16. Indicate how many of the following Intellectual Property Rights were applied for (give number in each box).	Trademark	
	Registered design	
	Other	

17. How many spin-off companies were created / are planned as a direct result of the project?	
Indicate the approximate number of additional jobs in these companies:	

18. Please indicate whether your project has a potential impact on employment, in comparison with the situation before your project:

❑ Increase in employment, or	❑ In small & medium-sized enterprises
❑ Safeguard employment, or	❑ In large companies
❑ Decrease in employment,	❑ None of the above / not relevant to the project
❑ Difficult to estimate / not possible to quantify	

19. For your project partnership please estimate the employment effect resulting directly from your participation in Full Time Equivalent (*FTE = one person working fulltime for a year*) jobs:	*Indicate figure:*

[10] Open Access is defined as free of charge access for anyone via Internet.
[11] For instance: classification for security project.

Difficult to estimate / not possible to quantify	❑

I Media and Communication to the general public

20. As part of the project, were any of the beneficiaries professionals in communication or media relations?

○ Yes ○ No

21. As part of the project, have any beneficiaries received professional media / communication training / advice to improve communication with the general public?

○ Yes ○ No

22 Which of the following have been used to communicate information about your project to the general public, or have resulted from your project?

❑	Press Release	❑	Coverage in specialist press
❑	Media briefing	❑	Coverage in general (non-specialist) press
❑	TV coverage / report	❑	Coverage in national press
❑	Radio coverage / report	❑	Coverage in international press
❑	Brochures /posters / flyers	❑	Website for the general public / internet
❑	DVD /Film /Multimedia	❑	Event targeting general public (festival, conference, exhibition, science café)

23 In which languages are the information products for the general public produced?

❑	Language of the coordinator	❑	English
❑	Other language(s)		

***Question F-10*:** Classification of Scientific Disciplines according to the Frascati Manual 2002 (Proposed Standard Practice for Surveys on Research and Experimental Development, OECD 2002):

FIELDS OF SCIENCE AND TECHNOLOGY

1. NATURAL SCIENCES
1.1 Mathematics and computer sciences [mathematics and other allied fields: computer sciences and other allied subjects (software development only; hardware development should be classified in the engineering fields)]
1.2 Physical sciences (astronomy and space sciences, physics and other allied subjects)
1.3 Chemical sciences (chemistry, other allied subjects)
1.4 Earth and related environmental sciences (geology, geophysics, mineralogy, physical geography and other geosciences, meteorology and other atmospheric sciences including climatic research, oceanography, vulcanology, palaeoecology, other allied sciences)
1.5 Biological sciences (biology, botany, bacteriology, microbiology, zoology, entomology, genetics, biochemistry, biophysics, other allied sciences, excluding clinical and veterinary sciences)

2 ENGINEERING AND TECHNOLOGY
2.1 Civil engineering (architecture engineering, building science and engineering, construction engineering, municipal and structural engineering and other allied subjects)
2.2 Electrical engineering, electronics [electrical engineering, electronics, communication engineering and systems, computer engineering (hardware only) and other allied subjects]
2.3. Other engineering sciences (such as chemical, aeronautical and space, mechanical, metallurgical and materials engineering, and their specialised subdivisions; forest products; applied sciences such as geodesy, industrial

chemistry, etc.; the science and technology of food production; specialised technologies of interdisciplinary fields, e.g. systems analysis, metallurgy, mining, textile technology and other applied subjects)

3. MEDICAL SCIENCES

3.1 Basic medicine (anatomy, cytology, physiology, genetics, pharmacy, pharmacology, toxicology, immunology and immunohaematology, clinical chemistry, clinical microbiology, pathology)

3.2 Clinical medicine (anaesthesiology, paediatrics, obstetrics and gynaecology, internal medicine, surgery, dentistry, neurology, psychiatry, radiology, therapeutics, otorhinolaryngology, ophthalmology)

3.3 Health sciences (public health services, social medicine, hygiene, nursing, epidemiology)

4. AGRICULTURAL SCIENCES

4.1 Agriculture, forestry, fisheries and allied sciences (agronomy, animal husbandry, fisheries, forestry, horticulture, other allied subjects)

4.2 Veterinary medicine

5. SOCIAL SCIENCES

5.1 Psychology

5.2 Economics

5.3 Educational sciences (education and training and other allied subjects)

5.4 Other social sciences [anthropology (social and cultural) and ethnology, demography, geography (human, economic and social), town and country planning, management, law, linguistics, political sciences, sociology, organisation and methods, miscellaneous social sciences and interdisciplinary , methodological and historical S1T activities relating to subjects in this group. Physical anthropology, physical geography and psychophysiology should normally be classified with the natural sciences].

6. HUMANITIES

6.1 History (history, prehistory and history, together with auxiliary historical disciplines such as archaeology, numismatics, palaeography, genealogy, etc.)

6.2 Languages and literature (ancient and modern)

6.3 Other humanities [philosophy (including the history of science and technology) arts, history of art, art criticism, painting, sculpture, musicology, dramatic art excluding artistic "research" of any kind, religion, theology, other fields and subjects pertaining to the humanities, methodological, historical and other S1T activities relating to the subjects in this group]

2. FINAL REPORT ON THE DISTRIBUTION OF THE EUROPEAN UNION FINANCIAL CONTRIBUTION

This report shall be submitted to the Commission within 30 days after receipt of the final payment of the European Union financial contribution.

Report on the distribution of the European Union financial contribution between beneficiaries

Name of beneficiary	Final amount of EU contribution per beneficiary in Euros
1.	
2.	
n	
Total	