Roofing slate standards: A critical review

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Highlights

- Roofing slate is the natural stone with the third-largest production by volume.
- There are several standards regarding slate, most important are EN 12326 and ASTM C406.
- The two most common test methods are water absorption and bending strength.
- Results may differ due to differences in the way tests are performed.

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Abstract

Roofing slate has its own regulations and standards, as with other natural stones. The two main markets for roofing slate are USA/Canada and Europe. Given the very long tradition of slate mining in these two areas, it is no surprise that the first regulations for roofing slate were developed there. In the USA and Canada, the technical requirements are compiled in the standard ASTM C406, while in Europe test methods and requirements are defined in EN 12326. There are also standards from China (GB/T 18600) and India (IS 6250), the emerging production countries in the slate market. This review article analyses and compares the current test methods for roofing slate. Also, new test methods are proposed in order to complement the information of the official standards.

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1. Introduction

Roofing slate is an important natural dimension stone, widely used all over the world since historical times. When properly installed, roofing slate can last hundreds of years. The oldest-known preserved slate roof is the Saxon chapel of Bradford-on-Avon, Wiltshire, England, built in the 8th century [1]. One of the first civilizations which used slate for roofing was the Romans, who developed an incipient industry in England, France and Belgium [2,3]. The use of slate was generally restricted to the geographic areas close to the deposits, but there is evidence of commercial transportation far away from the quarries [4]. However, it was during the Middle Age that slate became a popular roofing material for all types of buildings. Slate roofers’ schools flourished mainly in Belgium, southern Germany and northern France. On the other side of the Atlantic, the first slate quarry in the US was opened in 1734, in the Peach Bottom District of Pennsylvania [5,6], where slate quarrying was developed by Welsh immigrants [7]. Many of these miners settled in the slate-rich area of Vermont, but others moved to the other side of the continent. The use of slate roofs was quickly adopted along the East Coast of the US, and as in the case of Europe, mainly for special and characteristic buildings. With the consolidation of urban settlements, slate architecture became more and more important, but not nearly as widespread as in Europe. During the 20th century, the slate industry was severely affected by the two World Wars, as with the rest of the economy. In the 1960s, the new slate deposits from the NW of Spain began to produce a huge amount of cheap good-quality slate, forcing most of the slate quarries in the rest of Europe to close [8]. The technical advances in quarrying, with the introduction of diamond wire and the mechanization of the mills, drastically reduced the high rates of mortality among the miners and boosted production. The last years of the 20th century saw the consolidation of Spanish slate’s undeniable hegemony, which reached more than 80% of the world’s slate production. However, the recent economic crisis has hit the industry hard. Presently, Spanish hegemony is threatened by the ascendance of new production countries, such as Brazil, China, and India. These countries, especially China and India, have significant deposits of roofing slate, but still have a long way to go in terms of mechanization and quality control.

The growth of the slate industry brought along the need for the development of standards for the material. More than 95% of roofing slate is traded in Europe and the US, and so the standards in use on the market to certify slate are the European EN 12326 (parts 1 and 2), and the American ASTM C406, C120, C121 and C217. Other countries with important slate outcrops have their own standards, like the Chinese GB/T 18600 and the Indian IS 6250. Brazil, the second-largest producer in the world, does not have its own roofing slate standard as of yet.

1.1. ASTM roofing slate standard

The task of drafting the first standard specifically for roofing slates dates back to the 1920s [5], with the formation of the National Slate Association (NSA). The NSA was created by producers with the aim of regulating the market and unifying the criteria of production. At that time, slates were classified mainly by their physical appearance. The draft of the new standard took into account other features regarding weathering and durability. In July 1932 the Federal Specification Board approved the first draft of a slate standard, the specification SS-S-451 [5], a predecessor of the C406, the current ASTM standard for roofing slate, which was approved soon after. The ASTM’s first version was published in 1932, based on previous reports on slate. Early in the 20th century, Eckel published several results of research on roofing slates regarding the physicochemical characteristics of the rocks [9,10]. Dale et al. [11] and Nelson [12] published complete reports on the slate deposits of the USA, with a practical bias focused on mining and economic approach. Bowles [1,13] published exhaustive research on the characteristics of slates, discussing many of the issues that still affect the slate industry today, such as weathering, chemical composition and durability. At this point, the roofing slate industry in the USA was growing, becoming more mechanized and thus increasing its production. However, there were still no regulations or standards. The C406 standard classified the quality of slates according to the results of three test methods, ASTM C217 Depth of Softening, ASTM C121 Water Absorption, and ASTM C120 Modulus of Rupture. ASTM C406 introduced the classification of slate into grades (A, B and C) based on their weathering performance. In the late 1950s, the concept of service life was introduced with the new S1, S2 and S3 grades. Hence, for S1 the expected service life is over 75 years, for S2 from 40 to 75 years, and for S3, from 20 to 40 years.

Nowadays, ASTM C406 is reviewed, and if necessary revised, every five years by the C18 Dimension Stone Committee, or sooner if need be. In recent years, new changes have been made to C120, now named Flexure Testing, in order to adapt the standard to the new market requirements [14]. The ASTM standards have a long service record, being widely accepted by the American and Canadian markets. However, for some authors there is a low correlation between this expected service life and the test results [7,15,16].

1.2. EN roofing slate standard

During the 20th century, European countries with a tradition of roofing slate developed their own standards. By the 1990s, six countries had slate standards: the United Kingdom (British Standard, BS), France (Norme Francaise, NF), Germany (Deutsches Institut für Normung, DIN), Spain (Una Norma Española, UNE), Belgium (Spécifications techniques unifiées—Eénegemaakte Technische Specificaties, STS), and Italy (Ente Nazionale Italiano di Unificazione, UNI). The NF and DIN standards were focused on establishing the minimum acceptance criteria for the use of a rock as roofing slate, while the UNE, UNI and BS standards also specified these minimum criteria while at the same time classifying the material according to its characteristics and probable use [17]. The development of the standards in each country was determined by the type of slates quarried and the national architectural styles. For example, in the former British standard BS-680, the Modulus of Rupture (MoR) is...
not compiled, since most of the British slates are rather thick and have good mechanical values over 70 MPa [18], so testing the MoR was not such a necessity in the UK, although some low and characteristic longitudinal moduli, combined with the nailing of the slates more or less in the middle, can lead to a great deal of breakage during roofing. In France, the potential of iron sulfide oxidation was an important issue, so the former French standard NF P32-301 emphasizes the determination of iron sulfides and their distribution over the slate tile. These former national standards were substituted by EN 12326 (parts 1 and 2), written by the Technical Committee CEN/TC 128 Roof covering products for discontinuous laying and products for wall cladding, EN 12326-2:2000 Slate and stone products for discontinuous roofing and cladding—Part 2: Methods of test, was first released in December of 1999 [19], while EN 12326-1:2004 Slate and stone for discontinuous roofing and cladding—Part 1: Product specification, was only published in 2004. In May 2006, according to the European Construction Products Directive (89/106/CEE and 93/68/CEE), the CE mark became mandatory for selling and buying roofing slate in the EU. The CE marking must be attached to the pallets, and gives information about the characteristics according to the tests of EN 12326-2. The latest published versions date from 2011 for EN 12326-2:2011 Slate and stone for discontinuous roofing and cladding—Part 2: Methods of test for slate and carbonate slate for discontinuous roofing and cladding—Part 1: Product specification.

The most important changes introduced in EN 12326 since its creation were the definition of slate depending on its original genetic processes, and a third part, in development, dealing specifically with schist and schistose stones used for roofing. The issue about the petrological type of the slate had an important political and economic component. The Brazilian roofing slates tried to enter the European market, for which they had to accomplish the CE mark requirements. From a petrological point of view these rocks are in the anchimetamorphic field [20], between diagenesis and low-grade metamorphism. They display an incipient slaty cleavage parallel to the sedimentation layering, mainly generated by lithostatic compression [21–23]. These rocks can be considered as anchimetamorphic slates, or simply low-grade slates. This fact made part of the European producers claim that these roofing slates would not meet the EN 12326 requirements. The discussion ended with a modification in the definition of roofing slate in EN 12326, whereby Brazilian roofing slate became fully accepted in the scope of the standard. The TC 128 board signed a document in 2010 with the commitment of accepting Brazilian observers in any discussion affecting Brazilian materials [24].

EN 12326 represents a conscientious work of compilation of the most representative test methods from the former European standards, adapting them to the roofing slates of Europe as a whole. EN 12326 is still under development, and compiles important issues not reflected in ASTM, like the oxidation potential or the petrographic characterization.

### 1.3. Indian and Chinese roofing slate standards

The Indian IS 6250 and the Chinese GB/T 18600 standards are relatively new. Both China and India have used slate for roofing purposes since historical times, but this use was traditionally restricted to the areas close to slate outcrops [25]. In the last years of the past century, the slate industry of both countries has developed rapidly, due to the excellent economic expectations for the slate industry. However, the subsequent crisis from the beginning of the 21st century has slowed down this development. Their test methods are common to the other standards (Table 1). As with

| Table 1 Test methods compiled in the different roofing slate standards. |
|---------------------------------|-----------------|-----------------|-----------------|
|                                  | EN 12326         | ASTM C 406      | IS 6250          | GB/T 18600       |
| **Sampling**                     |                 |                 |                 |
| **Marking**                      | EN 12326-2 (4)  | ASTM C 406      | IS 6250-1981 (8) | GB/T 18600-2009 (6) |
| **Length/Width**                | EN 12326-2 (5)  | ASTM C 406      | IS 6250-1981 (9) | GB/T 18600-2009 (7) |
| **Straight edges**              | EN 12326-2 (6)  | ASTM C 406      | IS 6250-1981 (3) | GB/T 18600-2009 (4) |
| **Thickness**                   | EN 12326-2 (8)  | ASTM C 120      | IS 6250-1981 (8) | GB/T 18600-2009 (6) |
| **Flatness**                    | EN 12326-2 (9)  | ASTM C 121      | IS 6250-1981 (9) | GB/T 18600-2009 (7) |
| **Flexural strength (MoR)**     | EN 12326-2 (10) | ASTM C 120      | IS 6250-1981 (8) | GB/T 18600-2009 (6) |
| **Weathering behavior**         | EN 12326-2 (11) | ASTM C 121      | IS 6250-1981 (9) | GB/T 18600-2009 (7) |
| **Water absorption**            | EN 12326-2 (12) | ASTM C 406      | IS 6250-1981 (C) | GB/T 18600-2009 (D) |
| **Permeability**                | EN 12326-2 (13) | ASTM C 406      | IS 6250-1981 (C) | GB/T 18600-2009 (D) |
| **Freeze-thaw**                 | EN 12326-2 (14) | ASTM C 406      | IS 6250-1981 (C) | GB/T 18600-2009 (D) |
| **Thermal cycle**               | EN 12326-2 (15) | ASTM C 406      | IS 6250-1981 (C) | GB/T 18600-2009 (D) |
| **SO2 Exposure**                | EN 12326-2 (16) | ASTM C 217      | IS 4122-1967 (A) | GB/T 18600-2009 (D) |
| **SO2 Exposure, Weathering**    | EN 12326-2 (17) | ASTM C 406      | IS 6250-1981 (C) | GB/T 18600-2009 (D) |
| **Resistance**                  | EN 12326-2 (18) | ASTM C 406      | IS 6250-1981 (C) | GB/T 18600-2009 (D) |
| **Acid immersion**              | EN 12326-2 (19) | ASTM C 406      | IS 6250-1981 (C) | GB/T 18600-2009 (D) |
| **Non-carbonated carbon content** | EN 12326-2 (14) | ASTM C 406      | IS 6250-1981 (8) | GB/T 18600-2009 (4) |
| **Carbonate content**           | EN 12326-2 (15) | ASTM C 406      | IS 6250-1981 (8) | GB/T 18600-2009 (4) |
| **Specifications**               | EN 12326-1 (17) | ASTM C 406      | IS 6250-1981 (8) | GB/T 18600-2009 (4) |
| **Qualitative classification**  | NF 228° ATC*     | ASTM C 406      | IS 6250-1981 (8) | GB/T 18600-2009 (4) |
other national slate standards, the Indian standard has a clear influence from the characteristics of the slates quarried in India, being mainly focused on the slates used in the internal market. On the other hand, the Chinese standard is more internationally focused, compiling the main test methods from the EN and ASTM standards. It also defines the technical specifications required for the formats and quality control, with special emphasis on this last point.

1.4. Terminology

The definitions of roofing slate differ from one standard to another. EN 12326-1 makes a distinction between the petrological term slate, and the commercial term roofing slate, which comprises a wide group of rocks, like shale, slates s.s., schists, cinerites, siltstones, sandstones, and others [26,27], while ASTM C406 and IS 6250 do not make this differentiation. ASTM C406 uses the definitions from ASTM C119. Thus, in the words of the different standards, a slate is:

EN 12326-1:2014: a rock originating from clayey sedimentary rocks, including sediments of volcanoclastic origin and belonging petrographically to a range which begins at the boundary between sedimentary and metamorphic formation and ends at the epizonal-metamorphic phyllite formations. The predominant and most important components are the phyllosilicates and the cleavage resulting from a schistosity flux, caused by a low or very low grade of metamorphism. It is distinguished from sedimentary stones, which invariably splits along a bedding or sedimentation plane. The origin of the metamorphism can be due to tectonic or lithostatic compression, or a combination of the two.

ASTM C406: a microcrystalline metamorphic rock most commonly derived from shale and composed mostly of micas, chlorite, and quartz. The micaceous minerals have a subparallel orientation depending on their genetic process (tectonic or lithostatic compression) and the carbonate content. A third part of the standard is currently being prepared, dealing with schist and schistose stones. Due to differences between scientific and commercial criteria, these definitions have changed in almost every revision of the standard, although it seems that during the last revision a consensus was achieved. In any case, the definition of the different types of roofing slates according to EN 12326 is the most complex of all the standards, and is founded on a detailed petrographical analysis used to classify the rock.

2. Test methods

The tests can be divided into two main groups, Formatting and Technical Properties (Table 1). The first group deals with the standardized dimensions and tolerances that the slate tiles should have. These requirements are important, since today there are standardized slate roofing methods. If the slate tiles are not properly formatted, then it is difficult to install them. All the standards have specifications about the standardized dimensions of the slate tiles. ASTM C406, IS 6250 and GB/T 18600 indicate that the slate tiles should have a regular shape and dimensions, according to the standard format. EN 12326 goes a step further and has test methods referring to dimensions: straightness of the edges, rectangularity, thickness and flatness. The second group deals with the lifespan and performance of the slate, and it can be divided into three sub-groups: Mechanical Behavior, Weathering Behavior and Technical Characterization.

2.1. Mechanical behavior

The resistance to mechanical stress is measured by the Three Point Flexural Test, which gives the Modulus of Rupture (MoR). This test, with slight variations, is found in all the standards. The load is applied on the slate at a constant rate by a bar in the middle of two supporting bars (Fig. 1). At the line of load application, the upper part of the slate is subjected to compressive strength, while in the lower part it is subjected to tensile strength. The MoR represents the highest stress experienced within the material at its moment of rupture. The main characteristic of slate, the development of slaty cleavage, makes it strongly anisotropic. In order to check the influence of this anisotropy, slate tiles are tested in two directions, parallel and perpendicular to their grain or to Lineation L1, which is the structure formed by the intersection of the S0 sedimentation planes and S1 slaty cleavage planes. The orientation and angle of L1 is a key factor for MoR [29,30], and hence in the manufacturing of slate tiles [31]. Normally, to achieve the best mechanical performance, the tiles should be manufactured with their length parallel to the grain or to L1. This fact is well known by miners, but depending on the outcrop conditions this structure is not always easy to identify. Generally speaking, the MoR increases with the metamorphic degree. Shales usually have mean MoR values between 38 and 50 MPa [32], while slates, phyllites and schists have mean MoR values between 45 and 90 MPa. The general formula in all the standards used to calculate the MoR is \( R = (3 \cdot P \cdot l) / (2 \cdot b \cdot e^2) \), where \( R = \text{MoR (N/mm}^2) \), \( P = \text{failure load (N)} \), \( l = \text{distance between support bars (mm)} \), \( b = \text{width of sample (mm)} \), and \( e = \text{mean thickness of the sample (mm)} \). The MoR does not depend on the thickness of the slate; it is a value inherent to every slate variety. On the other hand, the effective load that a tile can support depends on its thickness, for example tiles for mountainous areas, with a heavy snow load, have to be thicker (and are generally rougher) than tiles used for other climates. The water content of the slate has been demonstrated to affect the MoR, since soaked slates have lower values of MoR than dry ones [31]. EN 12326-2 and IS 6250-1981 dry the
samples at 105–110 °C, while ASTM C120 and GB/T 18600 do it at 60 °C.

Another important point is the velocity of the load. For EN 12326, this is calculated taking into account the length and width (which are standardized) and the mean thickness, which is different for each sample. Normally, this value is between 600 and 900 N/min. For ASTM C120, the load velocity cannot exceed 5000 N/min. The load velocity influences the MoR results: for higher velocities this value is higher, since the applied strain is not dissipated, making the failure strength higher. The MoR values in ASTM C120 are therefore influenced by the thickness of the sample.

Some authors disagree with the usefulness of this test. A slate roof is not designed to have anyone walk on it, except the slater on occasion when making repairs [33], so there is no need to measure MoR, since the tiles are not going to support weight. Besides, there is no clear relationship between MoR and the expected service life [15,16,31]. The number of specimens measured for EN 12326-1 is at least 20, while for ASTM C120 it is at least 10. It is not clear if representative data are obtained by measuring this minimum number of slates. The MoR results are highly influenced by the angle between the sedimentation $S_0$ and the slaty cleavage $S_1$ [30] and sometimes by the number of Freeze–Thaw cycles [34]. This can cause legal issues, since the values declared by the manufacturer might not be the same after just a few years, depending on the climatic conditions. Despite all these issues, the MoR is widely used and understood in the slate world, constituting an important quality criterion.

### 2.2. Weathering behavior

There are two main types of weathering affecting roofing slates: physical and chemical. Physical weathering refers to the changes generated in the structure of the slate, while chemical changes cause the dissolution of existent minerals and the formation of new ones. Physical weathering is estimated through the measurement of the Water Absorption (WA), the resistance to Freeze–Thaw (FT), and Thermal Cycling (TC), while chemical weathering is...
measured by the Thermal Cycle (TC) and Sulfur Dioxide Exposure (SDE) tests. Compared to other rocks, roofing slate is not particularly prone to develop alterations, due to the low chemical reactivity of the main components (quartz and phyllosilicates) under normal atmospheric conditions. The most common chemical weathering affecting roofing slate is the oxidation of iron sulfides and the gypsumification of carbonates [35] (Fig. 2). Generally speaking, these pathologies are triggered in acidic environments, in the presence of water.

2.2.1. Water absorption and permeability

The Water Absorption (WA) test is the only test common to all the current and former roofing slate standards. The WA reflects the capacity of slate to absorb and store water. Water content is a crucial factor in weathering processes. It is generally accepted that the WA is a key factor for slate’s performance. The basis for this test is relatively simple, the more a slate absorbs water the higher its vulnerability to weathering under freeze-thaw circumstances. However, for some authors this correlation is not so clear, since certain slates may display high WA values but have proven high durability and high values of MoR [36]. The WA has also found to vary over successive cycles of wetting-drying, but with an erratic tendency. For [37], the general tendency is to increase the WA values over the cycles, as a result of the ageing caused by the test itself. On the other hand, for [34] the tendency is to decrease the values of WA, in this case explained as an effect of the loss of the interstitial water of the phyllosilicate minerals. It should be mentioned that these two studies both used the WA test method according to EN 12326-2, part 11, but using different slate samples – British slate for the first and Spanish slate for the second – which might explain the opposite results found in these two studies. In general, roofing slates present low WA (<0.5%), although this value depends largely on the test method. For ASTM C121 and GB/T 18600, the drying temperature is 60 °C, while for EN 12326-2 and IS 6250 the temperature is 105–110 °C. Originally, the ASTM dried the slates at 100 °C [31], but this temperature was changed in the following revisions. This difference in drying temperatures has an important effect on the results. A quick survey of WA values from ASTM and EN standards provided by production companies highlights that EN results are always higher than ASTM results. The influence of the temperature on the WA test can be explained by the performance of the phyllosilicate minerals (mica and chlorite). At 60 °C, the interstitial water is released from the slate structure, but at 110 °C a part of the adsorbed water in the mica minerals is also released. This water does not really play any role in the WA of the slate, since it forms part of the slate itself, and under normal conditions will never be released. The maximum temperature recorded by black slate exposed to direct sunlight during eight consecutive years was 72.74 °C [34]. Also, WA tests on other natural rocks are performed at temperatures of 70–60 °C (e.g. EN 1925 or EN 13755), it being widely accepted that higher temperatures modify the structure of the rock. The 110 °C drying temperature is only present in the EN 12326-2 standard, and it is a legacy of the former European standards that used this temperature (BS 680-1, UNE 232-191, DIN 52 204, and STS 34.03.06).

The Permeability test is only found in Indian standard IS 6250 and in former Italian standard UNI 8635. It consists in making a rectangular box using a slate tile for the bottom. The tile fits and is sealed perfectly on the box walls. The box is filled with water for 48 h, during which time the water may possibly leak through the slate itself. This test therefor measures the ability of slates to transmit water perpendicular to the split plane. Most of the roofing slate traded nowadays (i.e. shale, slate s.s., phyllite and schist) have inherent low permeability values, but other rocks such as sandstones and cinerites might have some fluid transmissivity on this plane, making it a useful test.

2.2.2. Freeze-thaw

The FT test is only found in EN 12326, and is performed if the WA value exceeds 0.6%. The justification of this test is the possible loss of integrity and resistance of slates with high WA values in cold regions. FT is a well-known weathering agent in all type of materials. When water freezes and forms crystals inside the rock, its volume increases by approximately 9%. This creates a crystallization stress on the rock bulk that might generate micro-cracking. Successive FT cycles may even disintegrate the rock. The aggressiveness of FT is directly related with the pore system of the material. Not all the pore sizes affect the integrity of the stone in the same way; the dangerous interval is between 0.1 μm and 10 μm [38]. Rocks with pore sizes below or above this range will suffer little from FT.

To perform the test, the slate samples are divided into two identical batches. One batch will act as a control, being subjected to the MoR test, while the other batch will be subjected to 50 cycles of FT. Each of these cycles consists of reaching a freeze temperature of –20 ± 2 °C for 5 h, and then 1 h of cooling at room temperature. However, some authors bring into question the accuracy of the MoR measurements before and after FT, since it is not possible to perform the MoR in the same sample twice, and there are known variations in BS even in tiles from the same slate slab [31]. There is not a clear trend in the MoR of slates in relation to successive FT cycles. The values for BS remain very similar, and in some cases even increase [34,39]. An internal report from a working task group of the TC128 came to the same conclusion. This result can be explained because rocks always have a certain amount of micro-cracks and a certain percentage of porosity. At the beginning of the (elastic) deformation, these cracks can be partly closed and the porosity reduced, leading to a change in the dimension and volume of the sample. If the stress is subsequently brought to zero, the whole deformation will be recovered, i.e. the slate will regain its former shape. This is in fact the defining characteristic of elastic deformation. The elastic behavior is characterized by the elastic constants Young’s modulus, bulk modulus, deformation modulus and Poisson’s ratio. The style of installation and the climatic region also play an important role in the damage induced by FT. Generally speaking, FT is accepted to cause no or little damage to roofing slates. Kessler and Sligh performed up to 2436 FT cycles in roofing slates without finding any structural damage or loss of integrity [31]. For slate, most of the pore system is below 0.1 μm [34], which is out of the critical pore range.

2.2.3. Thermal cycle

The TC is used to determine oxidation, staining, changes in the color of metallic inclusions, swelling, detachment, flaking or exfoliation (EN 12326-2:2011). The test method is common to other rocks (e.g. EN 16140), and forms part of all the slate standards except for ASTM C406, which does not specifically deal with oxidation features. It consists of submerging the sample in distilled water for 6 h at 23 °C, and afterwards heating the sample in a ventilated oven at 110 °C for 17 h. After this time there is 1 h of cooling at room temperature. These steps take a total of 24 h and form one cycle. The oxidation of the iron sulfides is favored by the conjugated effect of O2 release and temperature [35]. The main species of iron sulfides are (in order of abundance) pyrite, pyrrhotite, and marcasite. Pyrite and pyrrhotite do not have the same oxidation potential, with pyrrhotite much more weatherable than pyrite [40]. The oxidation reactions are favored by a low pH, and once triggered they contribute to diminishing the pH, generating SO2 and H2SO4, which may also attack the carbonates producing gypsumification. In the TC, during the immersion stage, the water acts as an environment for electron exchange, favoring the reaction of Fe2+ to Fe3+. Through the following heating stage, the dehydration temperature is reached, so the water adsorbed by the iron sulfide
The area of the tile in which these were allowed (Fig. 3). This classification tried to unify different test results into one index that is comparable to the life service index from ASTM C406. For IS 6250 and GB/T 18600, the test reports need only to describe the structural integrity of the slates, emphasizing the occurrence of weatherable minerals, i.e., iron sulfides. The results of the different sulfur exposure tests depend on the abundance and occurrence of weatherable minerals, i.e., iron sulfides and carbonates. The reagents used in the different standards are sulfur dioxide (SO$_2$) for EN 12326-2, and sulfuric acid (H$_2$SO$_4$) for IS 6250, ASTM C217 and GB/T 18600. In the EN 12326-2 test, samples are placed inside a hermetic container keeping a distance of about 10 cm with the SO$_2$ solution which evaporates. The 110 °C temperature reached during the thermal cycle is enough to trigger the superficial oxidation of the iron sulfides when coupled with water immersion. The main differences between the tests methods are the number of cycles and samples: EN 12326-2, 20 cycles, 6 samples; IS 6520-1981 16 cycles, 3 samples; and GB/T 18600, 25 cycles and 6 samples. Laboratory experience shows that between the 10th and the 15th cycle, the weatherable iron sulfides will have already shown alteration. The minimum number of cycles needed to have illustrative results can be set then at 15. Since the lowest number of cycles is 16 (IS 6520), the test results from these standards can be assumed as equivalents. The test report is also slightly different depending on the standards. EN 12326-1 gives a visual code (Fig. 3) according to the magnitude of the oxidation of the iron sulfides: T1 for no changes in appearance, surface oxidation of metallic minerals or color changes that neither affect the structure nor form streaks of discoloration; T2 for oxidation or appearance changes of the metallic inclusions with streaks of discoloration but without structural changes, and T3 for oxidation or appearance changes of metallic minerals that penetrate the slate and risk forming holes. A more detailed classification of these typologies was proposed [41], subdividing grade T1 depending on the mineralogy of the iron sulfides (pyrite or pyrrhotite), and redefining grades T2 and T3. This tentative classification tried to unify different test results into one index that is comparable to the life service index from ASTM C406. For IS 6250 and GB/T 18600, the test reports need only to describe the structural integrity of the slates, emphasizing the occurrence of oxidations. The TC was also compiled in the former BS 680, DIN 52-204 and STS 34 standards. For BS 680, the number of cycles was 15, while for DIN 52-204 it was 25. The test reports only had to mention the changes in slate integrity, as with IS 6250 and GB/T 18600. In France, the NF P 32-301 referred to the oxidizable iron sulfides as “iron pyrites” (pyrites de fer), specifying the area of the tile in which these were allowed (Fig. 3). This classification was adopted by GB/T 18600. A variation on the TC test method are the Thermal Sock (TS) and Soaking and Drying (SD) tests, by immersing the slate samples in the water without cooling. With this method the slate experiences a sudden change in temperature from 110 to 20 °C. This temperature change is used to determine slate integrity, in addition to weathering, and the results are expressed in variations of weight. Nowadays TS and SD are no longer used in any standard, but they were widely used in the preliminary reports for the ASTM [31,42], in the STS 34 and in the Spanish UNE 22-197. The TC has proved to be effective in predicting the oxidation potential of the iron sulfides, but has the disadvantage of taking from three to five weeks to complete, depending on the standard. The high temperature in TC affects the structure of the slate a great deal more than the FT, but in reality does not affect normal, good-quality slates.

2.2.4. SO$_2$ exposure, weather resistance and acid immersion

This group of tests has in common the use of sulfur solutions as a reagent. Each standard develops its own test method. In all cases, the objective is to check the weathering due to the interaction with a sulfurous atmosphere simulating urban and industrial environments. The carbonate present in the slate can react with the sulfur, forming gypsum (CaSO$_4$·2H$_2$O). The gypsum molecule has twice the size of the carbonate molecule, generating a stress that usually leads to the disintegration of the slate [31,35,39,43]. Urban environments, rich in S compounds, favor the gypsification processes. The presence of iron sulfides may also boost the weathering of carbonates [31]. These reactions are triggered in acidic environments, and the source of sulfur may be, besides pollution, the oxidation reactions of the iron sulfides. The results of the different sulfur exposure tests depend on the abundance and occurrence of weatherable minerals, i.e., iron sulfides and carbonates. The reagents used in the different standards are sulfur dioxide (SO$_2$) for EN 12326-2, and sulfuric acid (H$_2$SO$_4$) for IS 6250, ASTM C217 and GB/T 18600. In the EN 12326-2 test, samples are placed inside a hermetic container keeping a distance of about 10 cm with the SO$_2$ solution which is at the bottom. The proportion is 0.4% of solution per volume. Two different types of solutions are used, named A (25% SO$_2$ and 75% distilled water) and B (75% SO$_2$ and 25% distilled water). The number of samples is six, which are sawn into pairs. One set of pairs is immersed in water for 24 h, while the other set is dried in a ventilated oven at 110 °C for 24 h. Three dried pairs and three soaked pairs are put into a container with solution A, and the other six dried/soaked samples are put into a container with solution B for a total of 21 days. Afterwards, the slates are visually examined, searching for alterations, changes in color and detachments. A visual code is given (S1, S2 and S3) depending on the alterations developed. The nomenclature of this code is similar to the ASTM C406 lifespan code, which can lead to misunderstanding. The gypsification reaction requires a water supply, so wet environments boost the reaction rate [31]. However, this rate depends on other factors, such as the mineralogy, crystallization and abundance of carbonates, and it is possible
that under dry conditions the rate of gypsisification may be higher than under wet conditions, especially in urban environments. If the slate has a carbonate content higher than 20%, then for EN 12326-2 the slate has to pass a scratch test similar to the Weather Resistance (WR) from ASTM C217. The WR was proposed by Kessler and Sligh in 1932 [31], and finally established in 1948. It consists of submerging six slate samples in a 1% H₂SO₄ solution for seven days. Under these conditions, the whole integrity of the slate is compromised. The resistance of the slate is measured by the depth of a scratch made on the slate surface before and after submergence, using a special device. There is only one company supplying the necessary device, Taber Industries, as specified in ASTM C217. The scratch measurement must be accurate to 0.002 mm. This test procedure is the same in Chinese standard GB/T 18600. The IS 6250 test for acid exposure is simpler, just immersion of three slate samples in a 12.5% H₂SO₄ solution for 10 days followed by visual examination, looking for defects and detachments. No code is given to the slate, just a report of the possible defects developed.

2.3. Petrographical characterization

The petrographical characterization of roofing slate is only found in EN 12326, which uses the classic petrographical test methods (thin section examination and X-ray Diffraction, XRD) together with the quantification of the carbonates and non-carbonated carbon. The minerals found in roofing slates can be divided into three groups: main, secondary and accessory minerals. Main or primary minerals are those with abundances over 2%, used to classify the rock. In roofing slates these minerals are quartz, mica, chlorites, and sometimes feldspar and carbonates (mainly calcite). Quartz is the main component of many different rocks. Most of the quartz present in slate is of sedimentary origin. Mica and chlorites are phyllosilicates, a group of minerals with planar shape. In roofing slates, the most typical mica minerals are muscovite, sericite, illite, biotite and kaolinite, while for the chlorites the most typical are clinochlore and chlorite [27,31,37,44,45]. Feldspar is another common component in rocks, similar to quartz. The secondary minerals are the result of geological processes occurring after slate formation (diagenesis and metamorphism). These minerals are usually new crystals formed from the main minerals together with other minerals like iron sulfides (pyrite or pyrrhotite) and sometimes carbonates. They are easily identifiable by their shape and relationship with the rest of the slate bulk. Secondary minerals formed by diagenesis do not present traces of rotation or displacement, just a growth on the slate matrix, while secondary minerals formed during the metamorphism have cinematic traces [46]. Finally, the accessory minerals are those found in small proportions, below 2%. The most common minerals are rutile, leucoxene, tourmaline, hematite, chloroid, carbonates and iron sulfides. These minerals do not really affect slate quality (with the exception of iron sulfides and carbonates), and their occurrence merely reflects the former conditions of the original sedimentary basin. The Petrographical Examination (PE) is used to characterize the rock (nature, composition and internal arrangement), and, as pointed out before, is exclusive to EN 12326. ASTM C406 lacks this examination, even though in the preliminary reports that were used for the first drafts, the PE was used exhaustively [1,12,27]. In these works, the authors emphasized the validity of the data obtained using this technique. In 1926, the NSA even published a report in which there is a detailed table of the characteristics of slate minerals in PE [47]. However, and in spite of these works, the PE was never compiled in the ASTM norms [48]. The PE compiled in EN 12326-2 has its origins in the former standards: Spanish UNE 22-2001, Belgian STS 34, and German DIN 52-101 [49].

2.3.1. Microscopic examination

The PE, as used today in EN 12326, uses mainly microscopic examination with two different techniques, depending on the source of the light: light transmitted through a thin section, or light reflected from a polished section. Transmitted light microscopy is used for specimens that are relatively thin and semi-transparent, enabling a significant amount of light to pass through. In contrast, reflected light microscopy is mainly reserved for those specimens that remain opaque even when ground to a thickness of 30 µm or less. A transmitted light microscope has a light source below the microscope stage holding a thin section (25–30 µm thick) and sends light upwards through the thin section, up to the viewing point. A reflected light microscope has a light source above the sample and what is seen through the viewpoint are light waves reflected from the polished surface. Transmitted light is used to analyze the structural arrangement and mineralogy of the sample, while reflected light is useful to determine the metallic minerals (iron sulfides and iron oxides). In transmitted light, probably the most important attribute of the slate, its texture, is determined. Roofing slates usually display a lepidoblastic or porphyro- lepidoblastic texture [44], but other textures are frequent, such as granoblastic or porphyroblastic. The texture is the result of the geological processes that formed the slate, and is related to the fis-sility. Depending on the degree of recrystallization of the mica matrix, Dale classified slates into clay slate, in which the mica matrix does not present any or very faint aggregate polarization, and mica slate, in which the mica matrix has a strong and marked polarization [27], seen with a transmitted light optical microscope with crossed polarizer filters. A characteristic of the PE according to EN 12326-2 is the description and measurement of the mica layers, which are the result of the development of the schistosity. The morphological description of the mica layers was first proposed by [50], adapting the previous classification from [51]. The first version of EN 12326 adopted the petrographic analysis, adding the mica layers description and an index, the Mica Stacking Index (MSI), which quantifies the abundance and thickness of the mica layers using the formula $\text{MSI} = 10 \cdot \frac{m}{e}$, where $e$ is the average thickness of 10 mica layers in µm and $m$ is the number of mica layers per mm. This measurement has to be done at a 500× magnification with transmitted light. The MSI was developed from the petrological analysis of German roofing slates, which generally display good and regular mica plates, as with Spanish slates. A study performed on Spanish slates found a good correlation between the MSI and the BS [52]. However, there are other types of roofing slates with no clear mica planes but with good mechanical and splitting properties [49], like the slates from Penrhyn (UK) or the low-grade slates from Minas Gerais (Brazil). It is not possible to quantify the MSI in these roofing slates, due to their internal arrangement. Penrhyn slate has a characteristic porphyroblastic texture, with poor development of mica planes, while Brazilian slate does not present clear mica planes, since there is no full development of the slaty cleavage. Thus, it is difficult, or outright impossible, to measure the MSI in these two slates. When the MSI was adopted by EN 12326-2, it had to be applied to other materials that varied widely in petrological terms (shales, schists, siltstone, etc.) and differed from the German slates. For some of these new types, the MSI is difficult or impossible to calculate (Fig. 4).

Another characteristic of PE is the use of three sections along three perpendicular planes, the first (A) perpendicular to the slaty cleavage S₁ and parallel to the lineation L₁, the second (B) perpendicular to S₂ and L₂, and the third (C) parallel to S₁. The test report described in EN 12326-2 does not specify if each thin section has to be described separately, but in practice most of the petrographical test reports describe each thin section independently. This leads to repetitive descriptions, since sections A and B are virtually the
same, while for section C the information is usually very poor. The display of the mica levels in the C section is usually a mass of randomly oriented phyllosilicates, where it is not possible to describe other structures. Some authors [29] even point out that the traditional transmitted light microscopy has insufficient resolution to determine the arrangement of the mica levels, such that to achieve good results it is necessary to use Scanning Electron Microscopy (SEM). However, the effectiveness of thin section examination with transmitted light is beyond question, and applied to roofing slates it has proven to be useful and operative.

Reflected light, or Ore Microscopy, is especially used to determine the mineralogy of iron sulfides. As pointed out before, mineralogy (pyrite-pyrrhotite-marcasite) plays an important role in weathering and oxidation potential. This is an effective method, but in practice is not very useful, since the oxidation potential is already determined by the TC, and for the purchaser it is more important to know the oxidability rather than the species of iron sulfides present.

2.3.2. X-ray diffraction

In PE, X-ray Diffraction (XRD) is compiled as a characterization method in EN 12326-2. As for the microscopic examinations, XRD is a classic technique in Geology. EN 12326-2 considers two different methods of performing XRD, using powdered samples and polished slabs according to the direction of the cleavage. The powder method, then, is suitable to determine the mineralogy and the proportions of the minerals, while the polished slab method is used to determine the mineralogy influenced by the orientation in the sample. The diffraction of polished slabs should show the geological trace of the slate, making it possible to identify the original quarry of most types of slate. However, several reports have used this technique in order to determine the orientation and degree of crystallization of the mica minerals, though without linking this information to the original quarry. Previously unpublished reports have shown that the original quarry can be determined comparing the polished slab X-ray diffractograms from a sample with the reference from the quarry [53–55]. There are several works dealing with the determination of the provenance of a slate using combined techniques. J. Walsh proposed a combination of PE, XRD, SEM and X-ray Fluorescence (XRF) to determine the original quarry of Scottish slates [56–58]. Cárdenes et al. determined the source stratigraphic unit, not the quarry, in the Spanish roofing slates using XRD and XRF [59]. In all these studies, the characterization of minor and trace elements by XRF has proven to provide essential information. The XRD can be also used to determine the Crystallinity Index or Kubler Index (IC) [60]. This method measures the crystallinity of the illite, finding an index that places the sample in the sedimentary or metamorphic fields. For some roofing slates that are on the boundary between the different fields, the determination of this index is the only way to place them, and hence give them the correct petrological name.

2.3.3. Apparent calcium carbonate and non-carbonated carbon content

Besides the petrographical analysis, the former DIN 52-201 measured the proportions of CO₂, S, C and CaCO₃. These elements are major components of the two groups of weatherable minerals, iron sulfides and carbonates. In an average chemical composition of a roofing slate [45,59], S is only found in the iron sulfides, so it is a good proxy to estimate the expected amount of iron sulfides. However, S is found in different proportions in pyrite (FeS₂, 46.6% Fe and 53.4% S) and in pyrrhotite (Fe₇₈S₃₂, 62.3% Fe and 37.7% S), and other less common iron sulfides such as galena (lead sulfide), sphalerite (zinc sulfide) arsenopyrite (FeAsS, 34.3% Fe, 19.7% S, and 46% As) and chalcopyrite (CuFeS₂, 30.4% Fe, 34.9% S, and 34.6% Cu). Thus, depending on the iron sulfide minerals present in the sample, the calculated proportion of these iron sulfides may vary enormously. CO₂ and CaCO₃ are an indirect measure of the amount of carbonates in the roofing slate. The analysis of C, or non-carbonated organic carbon (also called organic matter) deals with a different issue. In some German slates the weathering of C might lead to the development of a new pore system [61,62].
affecting the integrity of the slate. EN 12326 inherited two of these determinations, apparent calcium carbonate (CaCO₃) and non-carbonated carbon (C) content, discarding S and CO₃. EN 12326-2 calculates the proportions of CaCO₃ and C using thermocatalytic decomposition, but other methods are acceptable when the laboratory proves the correlation between both methods. The mass percentage of CaCO₃ is calculated assuming all the carbonate is present as the calcium salt. This calculation assumes that the only species of carbonate present is calcite, but this is not in fact accurate. Carbonates in roofing slate are usually calcite (CaCO₃, 40% Ca and 60% C) but also ankerite (CaFeCO₃, 25.7% Ca, 35.8% Fe and 38.5% CO₃), and occasionally siderite (FeCO₃, 48.2% Fe and 51.8% CO₃) [63]. There are two possible sources for the carbonates, the depositional environment (limestone fragments, shells) and infiltration and deposition by meteoric water. This last source mainly generates ankerite, calcite and siderite “flowers” and can also be present in some faults, while for the first source the resulting carbonates (mainly calcite and occasionally dolomite) are embedded in the slate bulk, dispersed or in small layers. As for the iron sulfides, the different proportions of the elements in each carbonate mineral makes the EN 12326-2 calculations inexact. Ankerite is easily recognizable because it turns red with weathering. C is usually present in two different ways, in layers or ribbons, of carbonate occurrence with ellipsoidal shapes that might reach up to 10 cm (Fig. 3), or also forming ribbons in the slate bulk. Ankerite is easily recognizable because it turns red with weathering. C is usually present in two different ways, in layers or ribbons, or scattered over the slate matrix in rounded fragments of 1–10 μm. The first case is what is known among miners as burnt slate, and is the result of exceptional accumulation of organic matter of botanical origin in certain parts of the slate outcrop. It is easily recognizable because the slate appears with a bright, black lustrous patina, which hinders the task of splitting. C is closely linked with color [27,64], giving a dark tone to the slate. According to EN 12326-1, the amount of C should be equal to or less than 2%. Nevertheless, there are slates with C higher than 2% [64,65] which are normally used as roofing slates without any further problems. For the CaCO₃ content, EN 12326 just marks the distinction between roofing slates (20% CaCO₃) and carbonate roofing slates (>20% CaCO₃), which require supplementary weathering tests.

### 3. Quality assessment

The final objective of any standard is to give information about the performance of the product under normal conditions of use. ASTM C406 and EN 12326 both offer an estimate of the performance of the slate, but using different concepts. As pointed out before, ASTM uses the results of three test methods to estimate a potential lifespan of the slate based on three grades. This classification is easily understood and used by the market. On the other hand, in Europe no service life is given, but the slate must attain the results should be submitted by the “purchaser or his authorized representative”. However, in reality, most of the samples are submitted by the producer. Obviously, the producer always tends to send the best-looking slates to the laboratory. The NF and ATG certification systems only use samples taken from the purchaser’s or their authorized representative.

A bivariate correlation analysis of test results retrieved from the webpages of the producing and commercializing companies was performed (Tables 2 and 3) in order to check any possible relationship between the different parameters that control quality. For the EN 12326-2 results (Table 2) there are no clear correlations between the parameters. The strongest correlation (0.578) is found between the results of the MoR test in longitudinal and transversal orientations, which is to be expected. A slate with high or low MoR

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**Table 2** Spearman’s bivariate correlations for EN 12326-2 test results.

<table>
<thead>
<tr>
<th>BS Long</th>
<th>BS Trans</th>
<th>WA</th>
<th>C</th>
<th>CaCO₃</th>
</tr>
</thead>
<tbody>
<tr>
<td>N</td>
<td>104</td>
<td>102</td>
<td>48</td>
<td>80</td>
</tr>
<tr>
<td>Bending Strength (BS)</td>
<td>0.578 **</td>
<td>0.019 **</td>
<td>0.101</td>
<td>0.029</td>
</tr>
<tr>
<td>Water Absorption (WA)</td>
<td>0.638 **</td>
<td>0.019 **</td>
<td>0.101</td>
<td>0.029</td>
</tr>
<tr>
<td>Carbone content (C)</td>
<td>1</td>
<td>0.254</td>
<td>0.104</td>
<td>1</td>
</tr>
<tr>
<td>0.0116</td>
<td>0.080</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Table 3** Spearman’s bivariate correlations for ASTM test results.

<table>
<thead>
<tr>
<th>BS</th>
<th>WA</th>
<th>WR</th>
</tr>
</thead>
<tbody>
<tr>
<td>18</td>
<td>18</td>
<td>17</td>
</tr>
<tr>
<td>Water Absorption (WA)</td>
<td>0.638 **</td>
<td>0.112</td>
</tr>
<tr>
<td>N</td>
<td>1</td>
<td>17</td>
</tr>
</tbody>
</table>

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*Significant correlation at level 0.01 (bilateral). N is number of data.*

*Goedkeuring* in de Bouw (BUtgb) also has its own quality certification. Both certifications use the test methods of EN 12326-2, with more restricted compliment ranges. From a legal point of view, roofing slates only need the CE mark to be sold in Europe, but the markets of these countries demand the NF and ATG marks. The main reasons are that poor-quality oxidizing slate is able to obtain the CE mark, and that the average customer has no idea about what really constitutes quality. These countries have a long tradition of slate roofing and hence a significant amount of historical heritage built with roofing slate, so the authorities have developed their own parallel system of quality assurance. Nowadays, many companies have their test results available on their websites. An overview of these test results from both the ASTM C406 and EN 12326 standards reveals some interesting points. Almost every test result achieves the best possible results for each standard. This means that virtually all the slate sold on the market is of the best quality, and no second or third choices are being sold. This is obviously not correct; the market has a huge volume of sales of second and third quality slates. The reason for this is that the companies use the best test results found at a single quarry, even when different qualities might be produced from this quarry. For EN 12326, “sampling should preferably be carried out by the recipient or his representative in the presence of the supplier”, while in C406 the samples should be submitted by the “purchaser or his authorized representative”; however, in reality, most of the samples are submitted by the producer. Obviously, the producer always tends to send the best-looking slates to the laboratory. The NF and ATG certification systems only use samples taken from the purchaser or their authorized representative.
values in one orientation will probably have high or low values in the other orientation. However, this correlation is not as strong as should be expected. Another correlation (0.410) is found between the C content and the longitudinal MoR, which suggests that the C content might enhance in some way the MoR values in the longitudinal orientation. The C could absorb part of the strength applied to the slate tile during the test, acting like a kind of soap and increasing the MoR resistance. However, for the MoR in the transversal orientation there is no correlation. Regarding the MoR data, it has to be taken into account that if the slate tiles are not made with their length parallel to L1 or grain (cut on grain), the laboratory technician might have some trouble preparing the test specimens parallel and perpendicular to the grain, and then the results might not be accurate. Finally, there is a minor correlation (0.351) between the WA and the carbonate content. For the ASTM results (Table 3), there is a clear correlation (0.638) between the WA and the Weather Resistance (WR). This means that the carbonate content is directly proportionate to the water absorption. The correlations shown for the ASTM results are also clearer due to the numerical format of the test data. EN 12326-2 gives the data of two important tests, TC and SDE, as codes. The assignment of these codes depends to a great extent on the technician’s criteria, since there is no visual reference in EN 12326, and the interpretation of the results is therefore very subjective. On the other hand, the alterations produced in these two tests are visual, such that it is hard to quantify them. A possible method could be the measure of the WA before and after the test. The variation of WA was measured by Dale, finding a correlation between the grade of weathering and the increase of WA [27, 42]. Another possible method to quantify the effect of these tests is to apply image analysis, measuring the surface affected by the weathering [66].

4. Final considerations

This analysis of the current roofing slate standards brings to light some interesting conclusions. It is difficult to accurately reproduce the final usage conditions of the slate in the laboratory, but there are some test methods that have been proven effective throughout the years. For the mechanical features, the Modulus of Rupture test is a good approach, but it could be simplified in the number of specimens in order to reach a statistically significant value. By calculating the accumulated average after each test, it is possible to determine the exact number of specimens to be tested, saving time and efforts. For the weathering tests, the EN 12326 TC and SDE have proven to be a good approach to the environmental conditions, but their results should be numerically quantified. The code system is not sufficiently accurate. The way to quantify these tests could be by measuring differences in weight and in Water Absorption, or through image analysis. The ASTM makes a numerical quantification, which confirms the correlation between the Water Absorption and the weathering caused by the acid attack. Water Absorption has also proven to be a good index for weathering, and so is compiled in all the standards. On the other hand, test methods like carbonate determination and content of non-carbonated carbon are ultimately not very useful, since their results are embedded in the different weathering tests, in particular in the SO₂ Exposure test. Also, the Freeze-Thaw test has proven to have little effect in roofing slates. Finally, the EN 12326 petrological determination is an essential tool to correctly classify roofing slate, but it could be simplified by deleting the calculation of the mica levels and reducing the number of sections to be examined. This test should be compiled in all the standards, not just in EN 12326. Generally speaking, all the roofing slate standards could be made simpler, since they contain inherited test methods that do not, in fact, provide correct interpretable information.

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