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A NEW ACCELERATED LABORATORY TEST FOR THE ASSESSMENT OF THE DURABILITY OF MATERIALS WITH RESPECT TO SALT CRYSTALLIZATION

Barbara Lubelli^{1*}, and RILEM TC 271-ASC members

KEYWORDS

Salt weathering, test procedure, assessment methods, RILEM TC 271-ASC

ABSTRACT

The RILEM Technical Committee 271-ASC was set up in 2016 with the aim of developing an improved procedure for the assessment of the durability of porous building materials, such as brick and natural stone, against salt crystallization, accelerating the deterioration process without significantly altering its mechanism.

The test procedure developed by the TC 271-ASC proposes a new approach to salt crystallization tests. It starts from the consideration that it is necessary to accumulate a certain amount of salt to activate the damage. Thus salt damage can be seen as a process developing in two phases: accumulation and propagation. Based on this approach, a new salt crystallization test procedure has been defined, consisting of two phases: a first phase, in which salts are introduced in the material and accumulate close to the evaporation surface, followed by a second phase, in which damage propagates because of repeated dissolution and crystallization cycles induced by re-wetting with liquid water and by relative humidity (RH) changes.

In this paper the procedure is described and the reasons for the choices made are elucidated. The procedure has been tested on two types of limestone and, at the moment of writing, is being validated in a round robin test carried out on 9 different substrates and involving 11 laboratories. Based on the results of the round robin test, the procedure will be fine-tuned.

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1 INTRODUCTION

Salt crystallization is a major cause of damage in porous building materials (e.g. [1]–[3]). Despite extensive ongoing research in this field, the complexity of the problem has hindered the use of theoretical models for forecasting decay due to salt crystallization. Nowadays, in the practice of construction and conservation, the durability of porous building materials with respect to salt crystallization, when not well-known from past field experience, is mostly determined by accelerated ageing tests. However, despite the availability of a European standard (EN 12370) [4], three RILEM recommendations (RILEM 1980 [5], MS-A.1 [6], MS-A.2 [7]) and other guidelines (e.g. [8]), a commonly accepted testing protocol does not yet exist. As shown by the extensive literature review published by the TC 271-ASC [9], researchers are reluctant to use standard test procedures and often modify them or develop new ones. The use of different procedures hinders comparison between the results of different studies, as the procedure can significantly affect the results [10]. The main limitations of existing (standard) crystallization tests can be identified in the fact that they are either time consuming (e.g. [6]) or not realistically reproducing the transport and crystallization process (e.g. [4]). This may result in unrealistic damage types. Moreover, none of the existing standards prescribes an accurate, reliable and quantitative method or technique for monitoring damage development during the test. This complicates comparison between different results.

The RILEM Technical Committee 271-ASC (Accelerated laboratory test for the assessment of the durability of materials with respect to salt crystallization) was set-up in 2016 with the aim of overcoming the above-mentioned limitations by the development of an improved test procedure for the assessment of the behaviour of building materials with respect to salt crystallization.

The salt crystallization test developed by the TC 271-ASC proposes a new approach [11], different from existing salt crystallization tests, and derived from a common approach to the durability of reinforced concrete [12]. It starts from the consideration that it is necessary to accumulate a certain amount of salt, i.e., reach a certain degree of pore filling [13], before damage initiates. Salt damage can thus be seen as a process developing in two stages: accumulation and propagation (Figure 1).

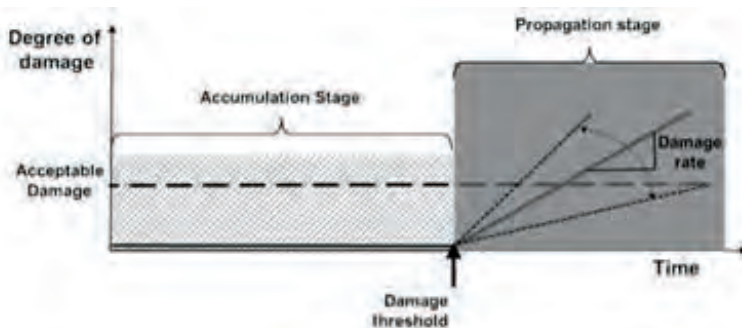


Figure 1: Schematic representation of durability of a stone, subject to salt damage, adapted from the concept proposed by Tuutti [12] to describe the durability of reinforced concrete (adapted from [11]).

In the past few years, an extensive experimental program has been carried out by the TC 271-ASC [14], supported by mathematical modelling of the transport and crystallization process [15]. Each variable (e.g., specimen size and number, salt content, contamination procedure, weathering cycles, assessment methods) has been the object of debate and research. The procedure has initially been tested on two types of limestone, Migné (FR) and Maastricht (NL), having very different porosity and pore size distribution [15]–[17]. At the moment of writing (Spring 2021), the procedure is being validated on nine different substrates (brick and natural stones) in a round robin test involving 11 laboratories.

In the following section the draft procedure is described, and details are provided on the reasons for the various choices made (*in italics*).

2 TEST PROCEDURE DESCRIPTION

2.1 Scope

The test procedure aims to reliably assess the durability of porous building materials against salt crystallization, accelerating the deterioration process without significantly altering its mechanism. Based on the results of the test, the user should be able to define a durability ranking of the different substrates with respect to salt crystallization.

The test reproduces the mechanism of salt damage triggered by capillary transport of salt solution towards the evaporative surface of a material. Damage by sea-salt spray is not considered.

The target group for the test are laboratories in the renovation and construction sectors. Therefore, feasibility has been a main requirement in the definition of the procedure; besides, assessment methods applicable without the need of sophisticated equipment or specialized expertise have been selected.

2.2 Salts

The test procedure considers damage due to sodium sulphate and sodium chloride, both tested as single salts.

These two salts have been selected as they are very common in the field and known for causing severe damage. Moreover, these salts significantly differ in their crystallization behaviour: sodium sulphate can crystallize in different forms, depending on temperature and Relative Humidity (RH) and its solubility is strongly affected by temperature. In contrast, sodium chloride can only crystallize as halite, at temperatures higher than 0°C, and its solubility is almost not affected by temperature.

2.3 Substrates

The procedure is (expected to be) suitable for brick and natural stone with an average open porosity of 15 vol% and more and Water Absorption Coefficient (WAC) values between 0.05-3 kg/m²s^{0.5}. Local heterogeneity within the samples and the average pore size distribution would require a critical view upon the results. For application on different materials (e.g., mortars) or brick and natural stones outside

the given range, as well as for combinations of materials, the procedure might require adaptations.

2.4 Specimen size, shape and number

The specimens consist of stone or brick cores of 50 ± 0.5 mm diameter and 50 ± 0.5 mm height. At least 3 specimens should be tested for each salt type and solution concentration.

This size is considered a good compromise between the need to reduce the size for speeding up the test and the necessity of specimens large enough to be representative of the substrate. Besides, this size would still be feasible, when adapting the procedure to assess combinations of materials, such as brick/stone/bedding mortar/pointing mortar/plaster/render.

The cylindrical shape has been preferred to the prismatic one, in order to avoid corner effects (i.e., potential stress concentrations at corners). Besides, the cylindrical shape facilitates sealing of the samples with paraffin film.

This minimum number of replicates is considered a good compromise between the requirement of taking the variation of the substrate into consideration and the necessity of keeping the test feasible, in terms of time and costs.

2.5 Preparation of the specimens

Cores should be drilled in such a way that minimum damage is inflicted to the substrate. Wet drilling or jet water cutting are recommended. In the case of brick, the surface usually exposed, i.e., stretcher or the header face of the brick, should preferably be the test surface. In the case of natural stone, the core drilling direction with respect to the bedding plane should be indicated in the test report. After drilling, brick or stone dust should be removed from the surface of the drilled cores, e.g., by the use of compressed air, water or a brush. The core specimens should be dried to constant mass in a (ventilated) oven at 40 ± 5 °C. Constant mass is reached when the difference between two successive weightings at an interval of (24 ± 2) h is $\leq 0.1\%$. The specimens should be sealed along their circumference with paraffin film, after pre-heating them in an oven at 50 °C for 10 minutes to achieve better adherence. The paraffin film should extend ca. 0.5 cm above the top surface of the specimens, in order to avoid that efflorescence and/or material debris, produced during the test, would fall down. Textile tape (water resistant and water vapour tight) can be used to secure the ends of the paraffin film and ensure sealing tightness during the entire test.

Paraffin film has been preferred to liquid paraffin or other impermeable resins, as it does not penetrate into the pores of the material, it can be easily removed and it is almost transparent.

2.6 Weathering procedure

The procedure consists of an accumulation and a propagation phase. The accumulation phase is identical for sodium sulphate and sodium chloride, except for the solution concentration. In contrast, the propagation phase varies depending on the salt type.

The reason for the differences is that the most effective damaging conditions (i.e., re-wetting and cycles of temperature and RH) have been selected for each salt.

Accumulation phase procedure

Preparation of salt solutions

Salt solutions should be prepared using demineralized water. Solutions can be prepared at room temperature. The following salt solution concentrations should be prepared:

- sodium sulphate: 1% and 5% (expressed as weight of Na₂SO₄/weight solution)
- sodium chloride: 5% and 10% (expressed as weight NaCl/weight solution)

The solution concentrations have been defined after a series of considerations supported by preliminary experiments, which are summarized hereafter.

It has been decided to use fixed solution concentrations (similarly to [4], [6], [7]), instead of fixed salt content in the specimens (expressed as weight salt/dry weight of specimen), as used e.g., in [18]. A fixed salt solution concentration is more likely to reproduce the field situation (e.g., rising damp of ground water with a given salt concentration) and it simplifies the procedure.

It has been decided to use more than one salt solution concentration. As the salt content in the specimen remains constant during the test, this choice enables a better ranking of the materials according to their resistance to salt decay. Moreover, it facilitates the estimation of the risks of future decay depending on the starting salt content, supporting thereby decision-making in the field of building conservation. The concentrations of the solution have been defined taking into account the following requirements:

- *High but still realistic salt content in the material. Based on field data, the maximum salt content has been indicatively defined as 3% (weigh salt/weight specimen);*
- *Realistic salt content but still high enough to cause damage. Based on data from previous experience [18], [19] and preliminary tests [14], minimum salt contents of about 0.5% and 1% (weight salt/dry weight specimen) have been defined for sodium sulphate and sodium chloride, respectively.*
- *Use of salt solution concentrations far from the saturation, in order to avoid immediate salt precipitation in the pores during contamination and favour salt transport and accumulation close to the evaporation surface.*

Contamination procedure

Before contamination, the specimen mass is stabilized for 4 hours at room conditions (22 ± 2 °C / 45 ± 15% RH). Contamination with salt solution occurs via capillary absorption through the bottom surface of the specimen, i.e., through the surface opposite to the evaporative surface.

The contamination procedure has been selected after testing different contamination procedures, shown schematically in Figure 2: 1) contamination from the top surface obtained by dissolution of the salt by hygroscopic adsorption, followed by

absorption of salt solution into the specimen; 2) simultaneous absorption of salt solution and drying (the so-called wick-effect, similarly to the procedure used e.g., in [20]); 3) capillary absorption of salt solution via the bottom surface, with or without additional rewetting with water from the bottom surface.

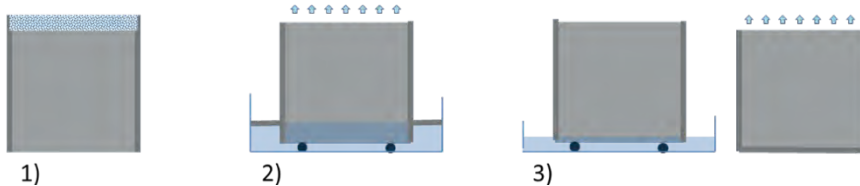


Figure 2: Schematic overview of the tested contamination procedures.

The following requirements were posed on the contamination procedure:

- Accumulation of salt in the outer layer of the specimen. Salt accumulation is necessary to obtain a high degree of pore filling and salt accumulation beneath the surface is resembling the field situation. Besides, salts present near the surface have a faster response to temperature and RH cycles, and this accelerates the damage process.
- Absence of efflorescence. Efflorescence decreases the amount of salt left in the pores, and thus potentially delays the damage process. Besides, efflorescence might result into slightly different salt content in the specimens at the start of the propagation phase, complicating thereby any comparison.
- No damage at the end of the accumulation phase. Because of the approach chosen, damage should initiate only in the propagation phase. Contamination and drying conditions should be selected in such a way to avoid the occurrence of damage during the accumulation phase.
- Ease of execution. The contamination procedure should be as simple as possible and not time-consuming

The capability of the different procedures to fulfil such requirements, as resulting from preliminary tests, is reported in Table 1 (see [14] for more details).

The mass of the salt solution used for salt contamination should be equal to the capillary moisture content (CMC) of the material [21]. This is determined as the point of intersection of best-fit straight lines drawn through the first and second stages of water absorption on a $t^{0.5}$ plot.

The use of the CMC guarantees that the top-surface (test surface) is wet at the start of the test and, thus, that evaporation occurs by liquid water transport to the surface, allowing for salt transport and accumulation in the outer layer of the material. The use of the CMC has been preferred to the water content at capillary saturation at atmospheric pressure, as for some materials the latest might take long to be reached, increasing thereby the risk of undesired efflorescence in the accumulation phase.

	From top surface by hygroscopic adsorption	From bottom surface, wick-action	From bottom surface, capillary absorption followed by drying	
			Without rewetting	With rewetting
Accumulation of salt in the outer layer of the specimen	-	+	++	+
Absence of efflorescence	++	+	+	-
No damage at the end of the accumulation phase	++	+	+	-
Ease of execution	++	+	+	+

Table 1: Comparison of the different contamination procedures, as resulting from preliminary tests. Scores: (-) it does not satisfy the requirement; (+) it partially satisfies the requirement; and (++) it fully satisfies the requirement.

Immediately after the full absorption of the salt solution, the bottom surface of the specimens is sealed with paraffin film and tape. The specimens are set to dry at 40°C / 15 ±5% RH, with very low air flow. In order to reduce the air flow, specimens can be placed in a box, closed with textile or Japanese paper (Figure 3), provided the required temperature and RH conditions are achieved. The temperature and RH should be monitored during the test.

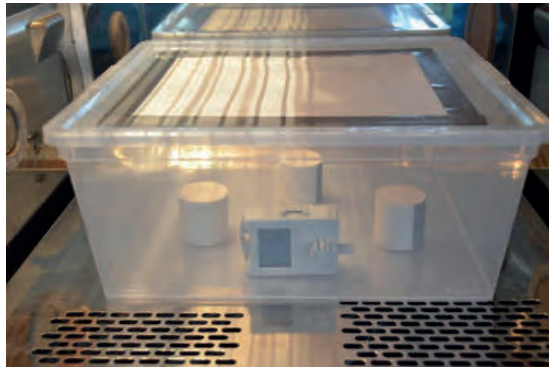


Figure 3: Example of specimens placed inside a box, the top of which is covered with a sheet of Japanese paper to limit air flow.

The drying temperature and RH have been selected after testing different drying conditions (20°C /15% RH, 20°C/50% RH, 40°C/15% RH and 40°C/50% RH) in terms of drying time, damage, efflorescence and salt distribution at the end of the drying.

It has been decided to use a very low air flow in order to limit as much as possible differences in air flow between different climatic cabinets and even at different location within the same cabinet.

The specimens should be weighted at each relevant step in the procedure and at regular intervals during drying. The accumulation phase ends when 80% or more of the introduced water is evaporated.

It has been decided to stop the drying when at least 80% of the introduced water is evaporated instead of waiting for full drying, which may take too long for some substrates. Moreover, this last phase is of little use for the accumulation of salts, as drying is takes place mainly by vapour water transport.

At the end of the drying period, the presence of damage and/or efflorescence is photographically recorded and described according to the methodology reported in 2.7.1.

Propagation phase procedure

Sodium sulphate

The propagation phase consists of 4 cycles, each of duration of 2 weeks. Each 2-week cycle consists of:

- 4 h cooling of the specimens at room conditions ($T=22^{\circ}\text{C} \pm 2$ at 45 % RH \pm 15%) during 4 h. Removal of the sealing from the bottom surface.
- Rewetting with water (80% of the initial water weight) by capillarity from the bottom surface at room conditions ($T=22^{\circ}\text{C} \pm 2$ at 45 % RH \pm 15%). Sealing of the bottom surface with paraffin film.
- Drying at room conditions ($T=22^{\circ}\text{C} \pm 2$ at 45 % RH \pm 15%) up to 24 h from the start of the re-wetting.
- Drying for 312 h (13 days) at $40^{\circ}\text{C} / 15 \pm 5\%$ RH

The weight of the specimens should be recorded at least at the end of each 2-week cycle.

The re-wetting by liquid water, carried out at room temperature, has been selected as it is known to be particularly effective in producing damage; in fact, rewetting with liquid water leads to dissolution of thenardite and immediate re-crystallization of mirabilite at high supersaturation, with consequent (severe) damage (e.g. [22], [23]). In order to facilitate the recrystallization of mirabilite, the temperature should be lower than 32°C . Because of this reason, the specimens are left to equilibrate at room temperature before rewetting and are stored at room temperature for 24 hours after re-wetting. The drying period has been defined by taking into account both the time needed for drying and the feasibility of the test procedure (periods different than multiples of 1 week, would require presence in the lab during the weekend).

Sodium chloride

The propagation phase of specimens contaminated with NaCl consists of 3 cycles, each cycle has a duration of 3 weeks. Each 3-week cycle (Figure 4) consists of:

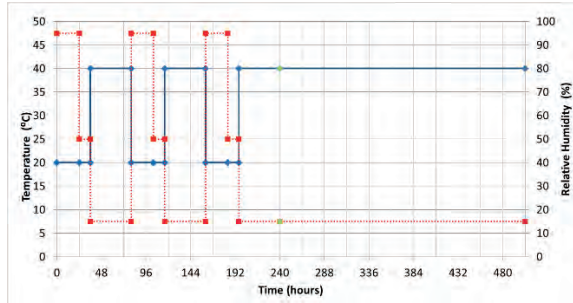


Figure 4: Scheme of the propagation phase for NaCl contaminated specimens.

- 24 h hygroscopic adsorption at 20°C/95% RH
- 12 h drying at 20°C 50% RH
- 44 h drying at 40°C 15±5% RH
- 24 h hygroscopic adsorption at 20°C/95% RH
- 12 h drying at 20°C 50% RH
- 44 h drying at 40°C 15±5% RH
- 24 h hygroscopic adsorption at 20°C/95% RH
- 12 h drying at 20°C 50% RH
- 44 h drying at 40°C 15±5% RH
- 4 h cooling at room conditions ($22 \pm 2^\circ\text{C} / 45 \pm 15\% \text{RH}$). Removal of the sealing from the bottom surface.
- Rewetting with water from the bottom surface with 50% water amount used for the initial contamination. Sealing of the bottom surface with paraffin film.
- 1 h storage of the specimens at RH > 95% (in order to allow for salt dissolution)
- 264 h drying at 40°C /15 ±5% RH.

The weight of the specimens should be recorded at least at the end of each 3-week cycle.

The use of rewetting cycles, both by RH cycles (leading to hygroscopic adsorption and dissolution followed by evaporation and crystallization) and by liquid water, has been selected as it is known that these can be particularly damaging for sodium chloride contaminated materials, much more than re-wetting cycles with liquid water [24], [25]. Moreover, the use of short RH cycles is effective to produce frequent dissolution/crystallization, without significantly mobilizing salts in depth. Besides, it does not introduce a large amount of water in the specimens, which would require long drying periods. However, some preliminary experiments showed that repeated RH cycles may favour the migration of the salts, accumulated in the outer layer, towards the inner core of the specimen (Figure 5). Because of this reason, rewetting with liquid water from the bottom surface of the specimen is introduced, to favour salt transport to the outer layer during drying. Storing the specimens at room temperature and high humidity conditions for 1 hour after re-wetting favours full dissolution of the salt.

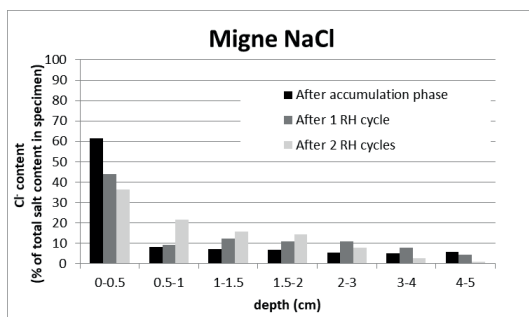


Figure 5: Chloride ion distribution (measured by ion chromatography) at end of the accumulation phase and after one and two RH cycles

2.7 Test report

Description of the damage

The development of the damage is assessed visually and photographically at the end of the accumulation phase and at the end of each 2- or 3-week cycle in the propagation phase. The occurrence of decay is photographically recorded. Pictures of each specimen are taken from different angles (perpendicular and with an angle of 45° with respect to the evaporation surface after each cycle, and also parallel to the surface at the end of the test, after removal of the parafilm and tape from the lateral surface). The type of damage is described according to the ICOMOS [26] and the MDCS atlases [27] and the extension and severity are evaluated according to a 4-point scale. The salt efflorescence morphology is reported as well (table 2).

This monitoring procedure has been chosen to allow for a simple but as much as possible objective assessment, which does not require special (laboratory) equipment and does not alter the specimens. Despite the attempt to standardize the damage description by the use of atlases, the assessment of the severity and extension of the damage, maintains a certain degree of subjectivity. Because of this reason, the quantification of the material loss at the end of the propagation phase is added to the procedure (see 2.7.2).

Material loss

The loss of material should be measured at the end of the propagation phase, after the drying phase of the last cycle, according to the procedure described hereafter. The surface of the specimens is brushed with a soft toothbrush, after removing the paraffin film wrapping. The debris (brushed from the surface and possibly including efflorescences) is dried at 40°C for 24h. The weight of the debris is recorded (d_1). The debris is put in a glass beaker with demineralized water for 24h at room conditions to dissolve the salt efflorescence. The amount of demineralized water used for dissolution should be 10 times the weight of the debris, in order to ensure full dissolution of the salts. The solution is filtered using a filter paper for medium/fast filtration. The weight of the dry filter paper is recorded (m_{fp}). After filtration, the filter paper and the material left in it are dried at 40°C until constant mass and the cumulative mass (filter paper + material) is recorded (m_{cum}). The material loss is calculated as follows:


Specimen code	Accumulation/propagation (cycle n.)	Photos	Damage type	Severity of damage	Extension of damage	Efflorescence type
Mi.1.Na-Cl	Propagation (cycle 3)		Loss of cohesion/Powdering	● (minor)	■ (25-50% of the surface)	No efflorescence

Table 2: Example of evaluation form.

$$\text{Material loss (d}_2\text{)} = m_{\text{cum}} - m_{\text{fp}} \quad (1)$$

The weight of the salt efflorescence is calculated as follows:

$$\text{Weight of salt efflorescence} = d_1 - d_2$$

This procedure for the quantification of the damage is somewhat more laborious and time consuming than recording the mass change of the specimen, as suggested by the European Standard 12370 [4]. This decision has been taken, as recording only the mass change of the specimen has the important limitation of not distinguishing between material loss and salt remaining in the pores.

3 DISCUSSION

The above-described procedure is the result of a 4-year long discussion and experimental work by the TC 271-ASC members. However, some points are still subject to debate, and will be clarified when the currently on-going round robin test is completed.

A first question is if the selected salt concentrations are effective for determining differences in damage and suitable for all substrates within the studied range, or if they should be fine-tuned according to the properties of the substrates.

Another question concerns the length of the drying period in the propagation phase cycle. This has been defined based on experiments on Migné and Maastricht limestones. The on-going round robin test will check this requirement for a wider range of substrates; based on the results, the water amount used for re-wetting and/or the length of drying period might be slightly modified.

A last point concerns the possible cementing effect of sodium chloride, which could keep the loose material together, leading to underestimation of the damage [19]. In the on-going round robin test, the cementing effect of sodium chloride is investigated at the end of the procedure by brushing specimens before and after slightly wetting their surface.

4 CONCLUSIONS

In this paper a new procedure for the assessment of the behaviour of porous building materials, such as brick and natural stone, under the influence of salt crystallization is described, aiming at overcoming (some of) the limitations of existing (standard) crystallization tests. This procedure consists of two phases: a first phase, in which salts are introduced in the material and accumulate close to the evaporation surface, and a second phase, in which damage propagates because of repeated dissolution and crystallization cycles, induced by re-wetting with liquid water and by RH changes.

The procedure has been developed and tested as effective on two substrates. At this moment, the procedure is being tested on 9 substrates in a round robin test involving 11 laboratories. Based on the results of the round-robin, the procedure will be fine-tuned. The TC 271-ASC expects to publish the procedure in 2022.

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