





# A comparison of methods to assess the resistance of reinforcing steel against chloride-induced corrosion in concrete—Particular consideration of 12% chromium steel

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## Funding information

Steeltec Group

## Abstract

The critical chloride content  $C_{\text{crit}}$  is an indicator of the corrosion resistance of reinforcing steels in concrete. In this article, several experimental methods for determining the critical chloride content  $C_{\text{crit}}$  of mild and alloyed (12% Cr) steel in cementitious materials are compared. The methods used include accelerated polarization tests and tests employing more natural (unpolarized) conditions in which chloride ingress occurs by diffusion and capillary uptake. The advantages and disadvantages of the methods are discussed, in particular, against the objective of reaching a compromise between applicability to practice and feasibility in laboratory testing.

## 1 | INTRODUCTION

The ability to reinforce steel embedded in concrete to withstand corrosion in chloride exposure environments can be expressed with the so-called critical chloride content ( $C_{\text{crit}}$ ), which is a concept that was originally proposed for carbon steel,<sup>[1–3]</sup> and later applied to stainless steels.<sup>[4,5]</sup> For stainless steels, the corrosion resistance can also be classified with the pitting resistance equivalent number (PREN).<sup>[6]</sup>

The  $C_{\text{crit}}$  is a measure for the chloride concentration in concrete at which corrosion of the reinforcement steel

starts. This parameter can be used for the qualitative comparison between different steel grades, such as for ranking steels according to their corrosion resistance in a given environment. Moreover, to probabilistically assess the effect of different steel grades on the life of a structure,  $C_{\text{crit}}$  is used as an input parameter in service life models.<sup>[7]</sup> However, it should be mentioned that such service lifetime predictions are very sensitive to the value of  $C_{\text{crit}}$ .<sup>[8,9]</sup> Therefore, accurate and practice-related measurements of  $C_{\text{crit}}$  are essential for making reliable service life predictions. Various methods have been proposed for the measurement of  $C_{\text{crit}}$  and many factors

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have been experimentally found to influence  $C_{\text{crit}}$ .<sup>[3]</sup> There is no consensus about the optimal method for the determination of  $C_{\text{crit}}$ , but many authors have pointed out that testing conditions should be representative of the conditions in reinforced concrete structures if the resulting  $C_{\text{crit}}$  were to be applied to engineering practice.<sup>[10]</sup> Thus, many of the proposed test methods, though modified from practical conditions, are based on studying different model systems, including a rebar segment, a surrounding cementitious matrix, and an approach to introduce chlorides.<sup>[3]</sup>

In contrast, the PREN solely is a sum of factors representing the amount of different alloying elements in the metal. These empirical factors are based on experience in pH neutral environment. Therefore, the PREN may be considered as a crude and fast method to rank different steel grades in such environments. However, its applicability to stainless steels in alkaline porous media such as concrete solution is questionable. Additionally, the PREN does not consider the microstructure or surface condition of the reinforcing steels, and the PREN ignores the influences related to the concrete and exposure conditions, which are all known to markedly affect the corrosion resistance of steel in concrete.<sup>[11]</sup> Moreover, the PREN does not allow for quantitative predictions of corrosion performance of stainless steels in a given environment. In particular, predictions for the time until corrosion initiates are impossible with the PREN concept. Another weakness of the PREN is that it does not allow comparing alloyed steels to mild carbon steel, which still is the most widely used reinforcing steel in practice

In this study, various methods to assess the corrosion resistance of reinforcing steel in concrete exposed to chlorides are compared. The different experimental approaches are applied to carbon steel (B500B, steel type 1.0439 according to the European standard<sup>[12]</sup>) and an alloyed chromium steel (12% of chromium, steel type 1.4003 according to Reference [12]). For all tests carried out by different laboratories described in this study, the steel grade 1.4003 was supplied from the company Steeltec Group (steel brand name “Top12”); for steel type 1.0439, local materials were used. These experiments give an overview of different methods to determine  $C_{\text{crit}}$  for mild and alloyed steels.

The most practice-related tests for the determination of  $C_{\text{crit}}$  are long-term exposure tests under conditions that comply with the exposure conditions during the service life of a reinforced concrete structure. However, determining  $C_{\text{crit}}$  requires the initiation of corrosion, which can take years or even decades in the case of stainless steel in concrete. Thus, this long experimental time is a severe limitation for laboratory testing. The consequence

is that  $C_{\text{crit}}$  tests have to be accelerated in one way or another. Various acceleration methods have been reported in the literature,<sup>[3]</sup> some of them (e.g., electrical migration techniques) being more efficient but less realistic than others (e.g., bulk diffusion). Here, we use a selection of methods that represent a wide range of test conditions. The effect of the testing conditions on the test result is discussed and an assessment of the different methods with respect to their applicability to practice is made.

## 2 | METHODS

In the following, the different test methods used in this study are briefly described. A summary of the relevant details can be found in Table 1. The methods will be referred to as *lollipop test* (Section 2.1), European Commission for International steel standardization (*ECISS test* (Section 2.2.), *potentiostatic polarization test* (Section 2.3), *capillary suction test* (Section 2.4), and *ETH method* (Section 2.5). Two of these methods are standardized (*ECISS test*<sup>[16,17]</sup> and *potentiostatic polarization test*<sup>[18]</sup>), and the *ETH method* has been published in full detail to be repeated by any laboratory<sup>[13]</sup> and widely used for  $C_{\text{crit}}$  testing.<sup>[19]</sup> The *lollipop test* and the *capillary suction test* are procedures adopted for single studies and are described below.

All tests were performed with steel type 1.4003 with the material composition given in Table 2. For steel type 1.0439, locally available products were used, which may differ slightly in composition.

### 2.1 | Lollipop test

Rebar samples with a length of 150 mm and a diameter of 10 or 12 mm were partly embedded in cylindrical mortar specimens with a diameter of 30 or 32 mm to obtain a cover thickness of 10 mm (see Figure 1a). The rebar samples were coated at two locations inside the mortar and on the outside top of the sample set-up to avoid ingress of chlorides from the sample top and to avoid corrosion of the cut rebar surface. The internal coating consisted of a cement paste layer covered by an epoxy resin layer. The rebar length exposed to the chlorides was ca. 60 mm. The mortar consisted of a CEM I 52.5 R or a CEM II/B-M (S-T) 42.5 R (with slag and burned shale) and sand with a maximum grain size of 1 mm. The water-cement ratio was held at a relatively high value of 0.6 to increase the chloride ingress rate and thus to reduce testing time. The tested rebar types were as follows: (i) ordinary concrete rebar of hot-rolled carbon steel

TABLE 1 Different experimental setups. All experiments were conducted in a laboratory environment; the temperature was between 20°C and 25°C

Experimental setup	Number of specimens	Length and diameter of exposed steel (mm)	Chloride exposure	Detection of corrosion initiation (criteria of termination)	Chloride content measurement
Pitting resistance equivalent number	-	-	-	-	-
Lollipop	1.0439: 5 1.4003: 10	60; 10–12	Mortar samples in saturated NaCl <sup>-</sup> and lime solution; nonaccelerated	Potential drop of 100–300 mV	Total chloride content according to SN EN 14629 at rebar level (in between rips)
ECISS	1.4003: 30 1.4571: 50 1.4062: 30	60; 10	Mortar with mixed-in chlorides	Current density > 80 mA/m <sup>2</sup>	Not necessary
potentiostatic electrochemical test according to DIN EN 480–14	1.0439: 30 1.4003: 30	50; 8	Anodic polarization Mortar with mixed-in chlorides	Visual examination Current between 1 and 24 h of polarization is partial or permanently higher than the current of $t = 1$ h	Not necessary
Capillary suction	1.0439: 6 1.4039: 3 1.4039: 6 1.4003: 6 1.4003: 3 1.4003: 6	50; 10 50; 8 80; 8 50; 10 50; 8 80; 8	Anodic polarization Mortar samples in 5 M NaCl, pH-value 6.8, exchange of the solution every 2 weeks	Visual examination Potential drop > 100 mV	Chloride content in the phase boundary by laser-induced breakdown spectroscopy
ETH method on laboratory specimens <sup>[13,14]</sup>	1.0439: 9 1.4003: 9 1.0439: 10 1.4003: 6	1.0439: 60; 12 1.4003: 80; 12 1.0439: 60; 16 1.4003: 60; 14	Diffusion from the lower part of the sample, migration of chlorides with electrical field (0.4 A/m <sup>2</sup> referred to the concrete area)	Potential drop of at least 150 mV within 24 h and stability of lower potential for at least 7 days	Total chloride content with acidic dissolution and potentiometric titration according to codes <sup>[15]</sup> at the level of reinforcement
ETH method on structure <sup>[13,14]</sup>	1.0439: 18 1.4003: 18	100; 10	Application of chloride-containing solution at the cracked concrete	Durable corrosion current $\geq 5 \times$ passive current; potential drop of >100 mV, increasing with the next chloride-application	Total chloride content with acidic dissolution and photometry at the level of reinforcement

TABLE 2 Material composition for 1.4003

	C (%)	Si (%)	Mn (%)	P (%)	S (%)	Cr (%)	Mo (%)	Ni (%)	Al (%)	Cu (%)	Nb (%)	Ti (%)	V (%)	Fe (%)
1.4003 (8 mm)	0.02	0.73	0.53	0.02	0.00	12.32	0.05	0.53	0.01	0.04	<0.005	0.01	0.05	85.68
1.4003 (10 mm)	0.02	0.71	0.59	0.02	0.00	12.17	0.05	0.53	0.01	0.05	<0.005	0.01	0.05	85.78
1.4003 <sup>[12]</sup>	0.03	1.00	1.50	0.04	0.05	10.50-12.50	-	0.30-1.00	-	-	0.03	-	-	-

B500B/1.0439 ( $\varnothing = 10$  mm); (ii) Steel 1.4003 with rolling skin ( $\varnothing = 10$  mm); and (iii) Steel 1.4003 with well-pickled and well-cleaned surface ( $\varnothing = 12$  mm). The age of the pickled steel surface at the time of the first test series with CEM I was approximately 3 months.

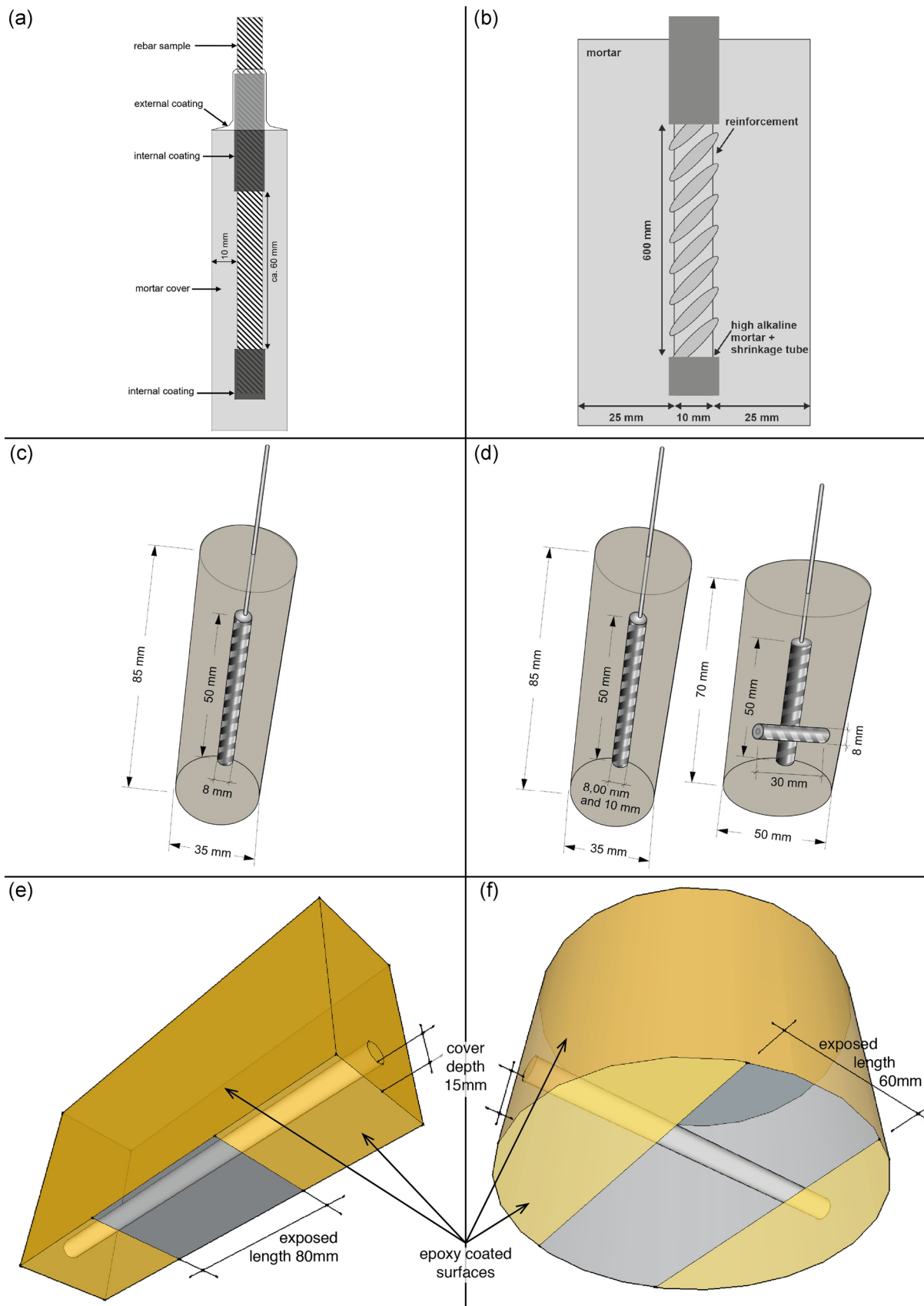
These lollipop samples were precured (passivated) in a saturated lime solution (without salt) for a duration of 7–98 days. After the preconditioning, the specimens were transferred to a saturated ( $\approx 5 M_{\text{NaCl}}/L$ )  $\text{NaCl}^-$  and lime solution at a constant temperature. The chlorides entered the specimens by diffusion. The passivation/depassivation state was monitored by continuous measurement of the open-circuit potential (OCP). The reference electrode for the measurements of potential was a saturated silver/silver chloride electrode (SSE). The onset of corrosion (depassivation) was indicated by a drop in potential (in the range of 100 to 300 mV). Within 1 day after the moment of depassivation, the samples were taken out of the solution and dried at 50°C for 2 h. Subsequently, the steel was detached from the mortar by splitting the sample into several parts. The steel and mortar surfaces were checked for corrosion signs under a microscope; rebars parts with corrosion pits or rust were photographed.

The chloride concentration surrounding the steel bars was measured from a powder sample that was collected by grinding off the rib texture on the mortar surface. The ribs contained between 1.2 and 1.6 g of mortar, a sample amount that was sufficient for chloride content measurement using XRF in compliance with a Swiss standard.<sup>[15]</sup> The chloride content measured is the weight fraction of the mortar material in the ribs. The used conversion factor for recalculating the measured chloride content to a weight fraction of the cement content was 2.75 (see Reference [20]).

## 2.2 | ECISS test

The *ECISS test* was developed for ECISS TC 104 WG3 (now CEN/TC459/SC 4/WG3) by Bertolini.<sup>[16,17]</sup> The aim of the *ECISS test* is to assess the  $C_{\text{crit}}$  for any higher alloyed (stainless) steels in a fast, reproducible and reliable, but not necessarily practice-related manner. Several studies proved its suitability considering multiple stainless steel grades.<sup>[17,21–23]</sup>

The procedure of the *ECISS test* is the following. The ribbed stainless steel rebar (diameter: 10 mm) is fixed centrally in a mortar cylinder with an exposed length of 60 mm and a mortar cover depth of 25 mm (see Figure 1b). The remaining steel surface is protected against crevice corrosion by the application of two different layers: first a layer of high alkaline mortar and



**FIGURE 1** Specimen geometry of the different experiments. (a) Lollipop, (b) ECISS, (c) potentiostatic electrochemical test according to DIN EN 480-14, (d) capillary suction, (e) ETH method on laboratory specimens, and (f) ETH method on specimens from a real structure [Color figure can be viewed at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)]



above a shrinkage tube. The composition of the mortar is: 450 g Portland cement CEM I 52,5R, 225 g distilled water, and 1350 g sand with a maximum grain size of 2 mm. The chloride concentration under investigation is dissolved in the mixing water.

The mortar specimens remain for 24 h in the mold before they are immersed in a saturated calcium hydroxide solution for 6 days at 23°C. After this curing period, a reference and a counter electrode (e.g., activated titanium oxide) complement the setup. Now, the free corrosion potential is recorded before each bar is polarized potentiostatically at 200 mV versus the saturated calomel electrode for 24 h in an anodic direction. The resulting polarization current is logged and is converted into the corrosion current density with regard to the rebar area exposed to the mortar. After the polarization period, the mortar sample is broken to visually inspect the steel surface for corrosion signs. One of the following two criteria must be fulfilled to assign the sample to “corrosion initiated”, respectively, the sample passes the test: (i) the maximum current density does not exceed 80 mA/m<sup>2</sup> and/or (ii) the steel does not show any visual signs of corrosion. Both criteria are explicit and simple. Thus, the outcome of the *ECISS test* is of binary nature, the sample pass (0) or fail (1).

The determination of the chloride threshold of particular stainless steel requires an iterative process. The procedure has to be repeated several times considering different concentrations of mixed-in chlorides in the mortar until 9 out of 10 samples pass the test. The aim is to identify the highest chloride concentration without corrosion initiation. As soon as the  $C_{crit}$  of particular stainless steel is known the *ECISS test* could serve as a quality control measure (declaration of performance).

For the sake of comparison three different stainless steel grades are considered for the assessment of the *ECISS test*, see Reference [23]: 1.4003 (PREN 12), 1.4571 (PREN 23), and 1.4062 (PREN 27). All samples were pickled and free of any signs of defects, such as oxides, cracks, rolling defects, and so forth.

### 2.3 | Potentiostatic electrochemical test according to DIN EN 480-14

In accordance with DIN EN 480-14,<sup>[18]</sup> two series of test specimens were produced for each chloride content to be tested. Series 1.0439 was produced with 8-mm reinforcing steel of type 1.0439 and series 1.4003 was produced with 8-mm ribbed reinforcing steel of stainless steel type 1.4003, which was pickled by the manufacturer. Figure 1c shows the structure of the test specimens.

Chloride contents of 0.0%, 0.5%, 1.0%, 1.5%, 2.0%, 2.3%, 2.6% and 3.0% by cem.wt. were admixed for Series 1.0439. For Series 1.4003 chloride contents of 0.0%, 0.5%, 1.0%, 1.5%, 2.0%, 2.3%, 2.6%, 3.0%, 3.5% and 4.0% by cem.wt. were admixed. Adding the NaCl content was carried out by a direct admixture into the mixing water. To determine the  $C_{crit}$  for the rolled surface of the rebars, the cutting edges of series 1.4003 were ground, pickled, and passivated.

Contrary to the normative specifications for the production of those test specimens, the material to be tested was not led out of the mortar cylinder and was not coated with a covering varnish in the transition area. The electron-conducting connection was carried out by a higher alloyed welding wire of 1.4571. This wire is connected to the base material by means of spot welding. After pickling and passivation, the spot-welded joint was assumed not to impair the corrosion resistance of the test specimen from series 1.4003 under the polarization conditions of this test. In the case of Series 1.4039, preinvestigations in 1 M NaOH solutions with sandblasted surface conditions were conducted.<sup>[24]</sup> The OCP of the 1.4571 wire is about 100 mV more negative than the OCP of the 1.0439 rebar under the same exposure. In both cases, no corrosion occurs in the area of the spot-welded joint under accelerated polarization in solution.

The mortar of the test specimens was produced according to DIN EN 196<sup>[25]</sup> with a 450 g CEM I 42.5 N as binder, 1.35 kg standard sand, and distilled water with a w/c-ratio of 0.5.

After manufacturing the test specimens, they were stored for 24 h at approx. 95% RH, afterward they were stripped of the formwork. Then the samples were stored in saturated calcium hydroxide solution for also 24 h. A three-electrode arrangement was used for the *polarization tests* with a high alloyed stainless steel serving as a counter electrode. A saturated calcium hydroxide solution was used as an electrolyte.

The specimens were polarized potentiostatically at 300 mV against a saturated SSE for 24 h. The resulting polarization current is logged and is converted into the corrosion current density regarding the rebar area exposed to the mortar. If corrosion is initiated during the polarization, this will be indicated by an increase in the measured current.

After the *polarization test*, the specimen must be broken, and the surface needs to be visually inspected for corrosion products. There are two criteria to pass the test, first, there are no corrosion products allowed at the surface and second, the measured corrosion current between  $t > 1$  h and  $t = 24$  h must be lower than the measured current at  $t = 1$  h.

## 2.4 | Capillary suction test in combination with highly local chloride measurements

*Suction tests* were also carried out at BAM to determine the  $C_{\text{crit}}$  without any polarization impact. For this purpose, varying test specimens were produced. The mortar composition corresponds to the described one in Section 2.3, without the addition of chloride. In addition, a series was produced that was carbonated in advance by an accelerated carbonation process. The carbonation chamber contains a  $\text{CO}_2$  content of 100% and an overpressure of 0.4 atmospheres. The aim is to adjust the pH value in the steel mortar phase boundary to values less than 8.6. The specimen design for the series with alkaline and carbonated mortar is shown in Figure 1d. The series 1.4003 and 1.4039 were prepared with three different sample variants of reinforcement dimensions. First, a single ribbed reinforcement rod with a length of 50 mm and a diameter of 10 mm. Second, same length but a diameter of 8 mm and third a reinforcement mesh cross with a diameter of 8 mm and a total length (addition of both parts) of 80 mm. With exception of the 8 mm rod samples (three samples), six specimens were prepared per sample variant. The surface condition of the Series 1.4003 is pickled by the manufacturer, the surface condition of series 1.0439 is as-received. Similar to Section 2.3, the electron-conducting connection was carried out by a higher alloyed welding wire of 1.4571. This wire is connected to the base material by means of spot welding. After pickling and passivation, the spot-welded joint was assumed not to impair the corrosion resistance of the test specimen from series 1.4003 because of the pronounced area ratio of the different steels. In the case of series 1.0439, the pronounced surface area ratio between rebar and stainless steel wire is so low that no effect on the corrosion performance of the rebar was expected. For the carbonated series with 1.0439 and 1.4003, the carbonation depth was evaluated by applying phenolphthalein at fresh fractures of unreinforced reference mortar prisms. This series just served as a carbonation reference to determine a successful carbonation process. The measured potentials after successful carbonation of the series with 1.0439 were used to verify the low pH value and the resulting depassivation of the reinforcement.

For the *suction test*, the samples were stored in a closed container with a 5 M NaCl solution. At the beginning of the exposure, the pH value of the solution was 6.8 and the solution was changed monthly. The samples are immersed at approx. three-fourths of the sample height. Before the start of exposure, the samples were dried for 24 h at 40°C, then left at room temperature for a further 24 h.

The potential of all samples was measured against a saturated Ag/AgCl electrode and was recorded over the duration of the exposure with a frequency of 1/60 Hz by a datalogger.

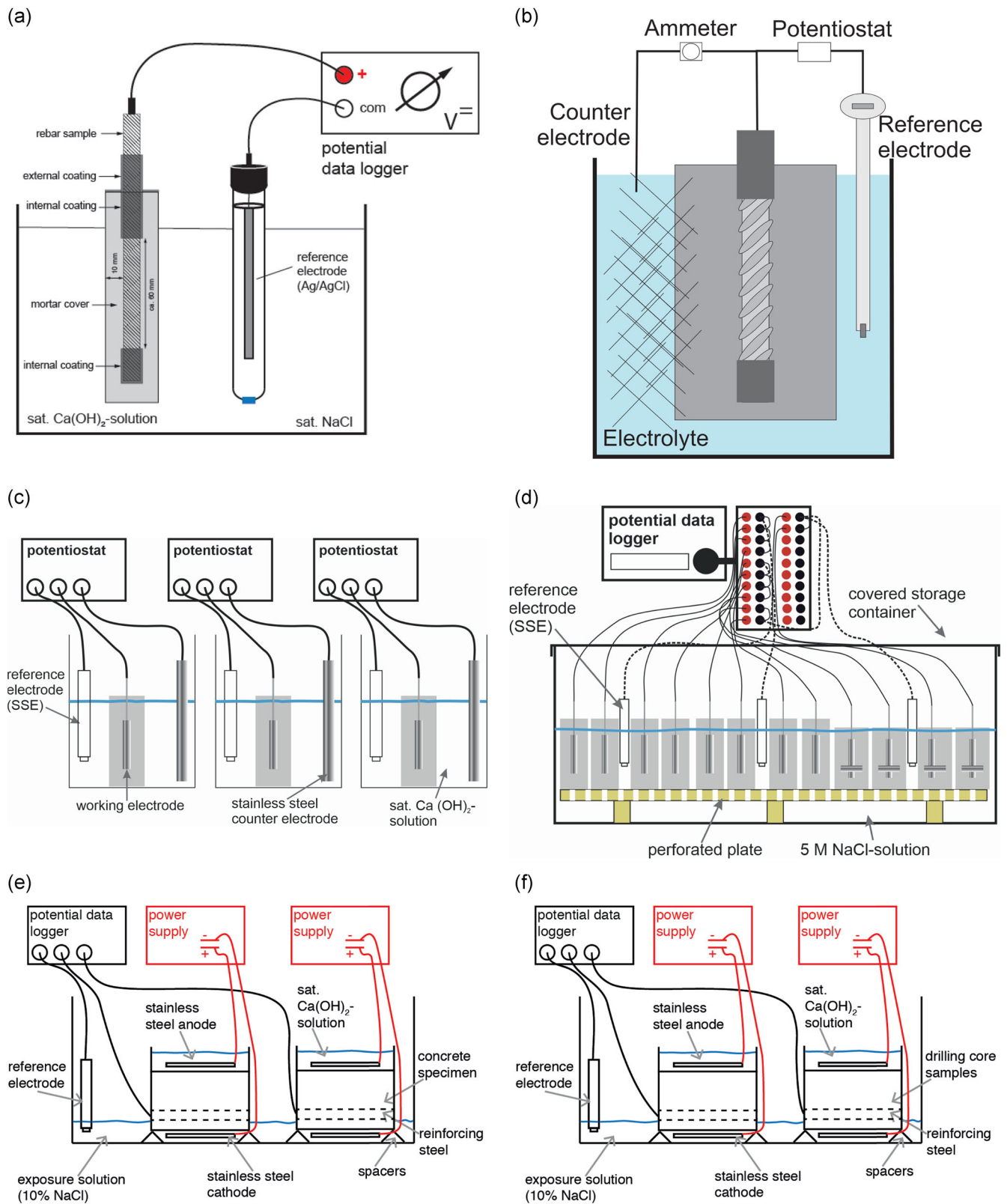
If the measured potential of a specimen has a drop of about 100 mV to lower values the specimen was removed from the exposure, split, and the corroded area was localized and marked as a point of interest (POI). Often the specimen did not show any corrosion products, so the surface of the exposed reinforcement had to be inspected for pitting corrosion by light microscopy. Afterward the chloride content of the mortar, in the range of the POI, was determined by laser-induced breakdown spectroscopy (LIBS).<sup>[26–30]</sup>

In LIBS measurement, a pulsed laser beam is focused on the sample surface, thereby vaporizing, atomizing, and ionizing sample material. The plasma induced by the laser emits element-specific radiation, which is analyzed spectroscopically. The resolution for the analyzed spots in the range of the POI was 1 by 1 mm. The FiberLIBS-lab system from SECOPTA analytics GmbH was used to investigate the chloride content of the POI. The quantification of the LIBS results was carried out using calibration samples with defined chloride contents and was carried out according to DIN 32645<sup>[31]</sup> or ISO 8466-1<sup>[32]</sup> and proofed for linearity with  $R^2$  of 0.993. A deviation between the two series of measurements could not be determined. A limit of quantification with 0.07% by cem.wt. and a limit of detection of 0.03% by cem.wt. were determined. Further information about the calibration samples and the fit are described in Reference [33].

## 2.5 | Moderately accelerated ETH method

This method consists in testing reinforced concrete samples, either retrieved from engineering structures or from laboratory-produced samples. In both cases, the samples undergo a number of specimen preparation steps before exposure and corrosion testing, as described in Reference [13]. In summary, cover depth was reduced to approx. 15 mm by cutting. To continuously measure the corrosion potential, a cable was connected to the reinforcement (screw connection). With an epoxy resin, both the lateral faces of the specimen and the end parts of the later exposed surface were coated (to avoid chloride ingress from these sides and to protect the rebar end parts from corroding) (see Figure 1e,f). Thus, the exposed rebar length is approx. 80 mm.

Subsequently, the specimens were exposed to chlorides by positioning the lower face of the specimens in a solution (Figure 2e,f). Initially, for the first week, the solution was chloride-free (tap water). After this, NaCl



**FIGURE 2** Exposure conditions of different experiments. (a) Lollipop experiments, (b) ECISS experiments, (c) potentiostatic electrochemical test according to DIN EN 480-14, (d) capillary suction-experiments, (e) ETH method on laboratory specimens, and (f) ETH method on specimens from a real structure [Color figure can be viewed at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)]



was added, starting from low concentrations that were after a few weeks stepwise increased, to reach a final concentration of 10% NaCl. This was typically achieved after 4 months. With an external saturated SSE placed in the solution, the potential of the steel was continuously measured to detect corrosion initiation. The criterion of initiation was defined as a potential drop for at least 150 mV that was maintained for 7 days.<sup>[13]</sup>

If the specimens were not initiated during this stage of chloride ingress by capillary suction and diffusion, the chloride transport was moderately accelerated by means of migration. An electrical field was applied across the concrete specimen, by positioning two external activated stainless steel perforated plate electrodes on both sides of the sample. One electrode was positioned on the lower side in the exposure solution; the other electrode was placed in a small pond mounted on the top surface (filled with saturated  $\text{Ca}(\text{OH})_2$ -solution). The applied current density was  $0.4 \text{ A/m}^2$  referred to the exposed surface area of the concrete specimen for 18 h each day. The potential was measured during the 6 h without the applied electrical field. The criterion for corrosion initiation was a decrease of 150 mV to the previous potential without an applied electrical field. No electrical field was applied after corrosion initiation and the potential was observed for 7 days.

After corrosion initiation, the specimens were removed from the exposure solution and the total chloride content was determined by cutting a thin disc of  $\pm 2 \text{ mm}$  with respect to the cover depth of the tested sample. The chloride content was measured with acid digestion and potentiometric titration.<sup>[15]</sup> The result was considered as the  $C_{\text{crit}}$  of the specimen tested.

Two different types of specimens were tested: specimens made in the laboratory and specimens retrieved from a bridge.

**Laboratory specimens:** Concrete specimens (beams  $20 \times 10 \times 5 \text{ cm}$ ) were cast with one centrally located, ribbed reinforcing steel bar (Figure 1e, ordinary Portland cement, w/c-ratio: 0.5, maximal aggregate diameter: 16 mm). One series was produced with steel grade 1.4003 (diameter 10 mm, pickled) and a second series was made with steel grade 1.0439 (diameter 12 mm, used in as-received condition). Per series, nine specimens were cast.

**Specimens from a structure:** Cores (diameter 150 mm) were drilled from a concrete bridge in the Swiss Alps (Sunnibergbrücke, constructed in 1997). The drilling cores contained centrally located reinforcing steel bars (Figure 1f). Ten specimens contained steel grade 1.0439 and six specimens contained steel grade 1.4003 (note that at the time of constructing the bridge, the 12% chromium steel was supplied in nonpickled condition). The method

of taking these samples from structures is described in more detail in References [13,14].

## 2.6 | Investigation on the depassivation behavior in cracked specimens exposed to chloride ions

Cracks are not considered in-depth in the context of this publication. Nevertheless, a short summary of investigations on cracked specimens shall be given here.

The target of the investigation on the corrosion behavior of 1.4003-rebars in chloride-containing cracks was to determine the chloride content within the cracks, which leads to a permanent depassivation of the rebars (1.4003 as well as 1.0439).

For the tests, reinforced concrete beams ( $l \times b \times h = 400 \times 100 \times 100 \text{ mm}^3$ ) with a central separating crack were produced. In total, 18 reinforced concrete samples with 1.4003 and 18 samples with unalloyed reinforcing steel 1.0439 as a reference, are examined. For the concrete design, a CEM I 42.5 N cement with a w/c ratio = 0.45 and an A/B 8 grading curve was used. The aspired crack width was 0.20 to 0.25 mm, which was achieved with median values of 0.20 mm for the specimens with 1.0439 rebars and 0.25 mm for the crack specimens with 1.4003. Chloride-containing solution with continuously increasing chloride concentration was regularly applied on the specimens. The aim was to determine the chloride content in the crack flank, which is linked with the first measurable stable corrosion initiation.

The depassivation of the rebars was determined by using titan-mixed-oxide ribbons either as counter electrodes or as reference electrodes (calibrated regularly against a CSE). As the free potential declined and the current increased, initiation of corrosion is indicated and the further chloride application was interrupted for 2 weeks. After stable corrosion initiation, the rebar was removed and the chloride content in the crack flank near the rebar was measured, which is defined as the  $C_{\text{crit, crack}}$ . In case of a repassivation of the rebar detectable by decreasing current to the passive corrosion current, the chloride application was continued by continuously increasing chloride concentration.

For further details in regard to experimental setups, see Table 1 and References [34–36].

## 3 | RESULTS

The tests described above lead to two different types of results and therefore, require different statistical treatments. Some of the tests determine the chloride content in the sample given that corrosion has initiated. Thus, the resulting data on  $C_{\text{crit}}$  is a continuous set of data that

can be represented in a cumulative frequency diagram. Other tests set different chloride contents and estimate whether corrosion initiation takes place or not. Here, the resulting data are a discrete data set of the chloride content with the binary outcome; the sample passed the test (0), so the steel remained passive, or the sample failed the test (1) due to apparent corrosion activity. Here, a binary regression based on the logit function is used to represent the statistical data set.

The results of these tests are given in Table 3 and graphically presented in Figure 3. The results are also graphically summarized in Figure 4.

### 3.1 | Lollipop test

The measured  $C_{crit}$  values for the tested steel types with CEM I are presented in Figure 3a. The curing time for passivating for these samples was between 7 and 98 days. For samples with CEM I, the average  $C_{crit}$  of the steel 1.4003 with the pickled surface was  $4.90 \pm 0.48\%$  chloride by cem.wt. The nonpickled steel 1.4003 (with rolled skin) had an average  $C_{crit}$  of  $2.05 \pm 0.30\%$  chloride by cem.wt. Under the same testing conditions, the average  $C_{crit}$  for ordinary, nonpickled carbon steel (1.0439) was  $1.25 \pm 0.53\%$  chloride

by cem.wt. The repetition of the experiments with CEM II and curing times of 212–244 days produced similar trends in results: For pickled steel 1.4003,  $C_{crit}$  was  $5.52 \pm 0.36\%$  chloride by cem.wt; for nonpickled steel 1.4003,  $C_{crit}$  was  $3.84 \pm 0.68\%$  chloride by cem.wt; and for ordinary, nonpickled carbon steel (1.0439),  $C_{crit}$  was  $1.76 \pm 0.47\%$  chloride by cem.wt. These *lollipop tests* suggest that the cement type and passivation time (or concrete age) have a moderate effect on  $C_{crit}$  (see Reference [11]).

### 3.2 | ECISS test

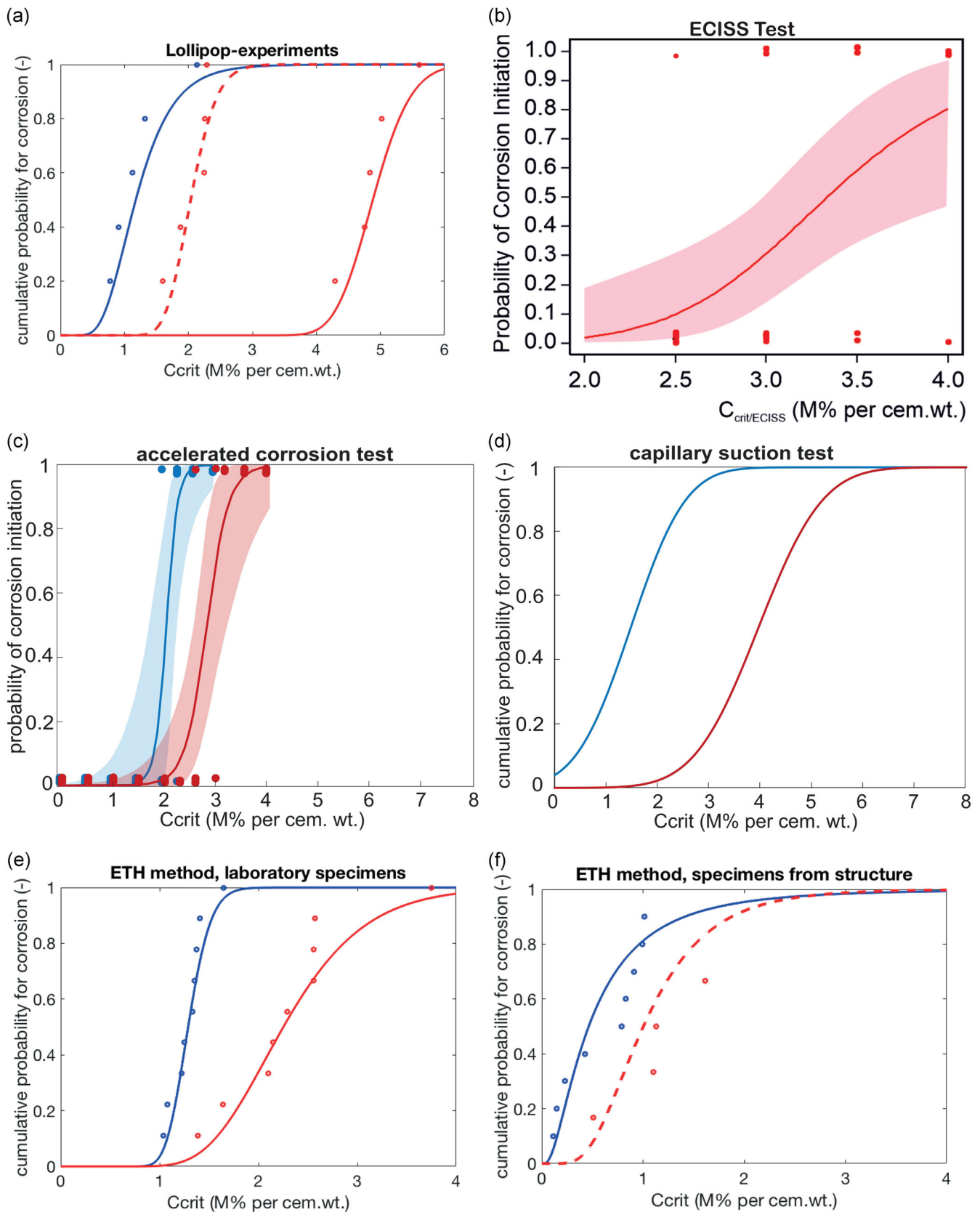
The determination of the chloride threshold value of particular stainless steel according to the *ECISS test* takes about 6–8 weeks without any prior knowledge of the  $C_{crit}$ . The results of the  $C_{crit}$  of the investigated stainless-steel grades were: 1.4003—2.5% chloride by cem.wt., 1.4571—5.5% chloride by cem.wt., and 1.4062—7.5% chloride by cem.wt. For this particular example, the  $C_{crit}$  increases with increasing pitting resistance equivalent number and the achieved  $C_{crit}/ECISS$  values cover a broad range for possible applications.

The *ECISS test* depends on discrete data to get binary outcomes. The discrete data is the chosen categories of

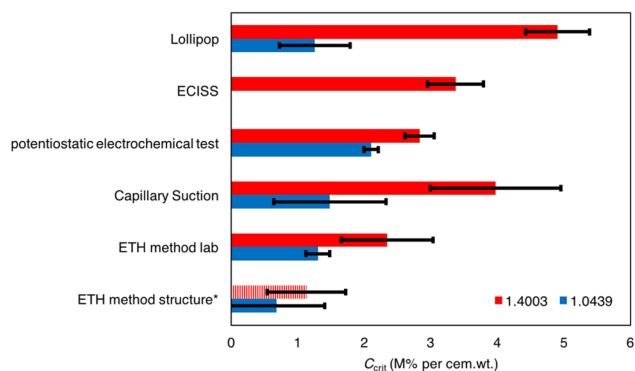
TABLE 3 Results of critical chloride content ( $C_{crit}$ )

Experimental setup	$C_{crit}$ (% by cem.wt.) 1.0439 mean value $\pm$ standard deviation	$C_{crit}$ (% by cem.wt.) 1.4003 mean value $\pm$ standard deviation	Remarks
Pitting resistance equivalent number	-	-	-
Lollipop	$1.25 \pm 0.53$	(Nonpickled: $2.05 \pm 0.30$ ) $4.90 \pm 0.48$	$n = 5$ for each series. Curing time = 7–98 days
ECISS	-	2.5 (10% corrosion probability) $3.37 \pm 0.42\%$	Binary regression (logit function)
Potentiostatic electrochemical test according to DIN EN 480-14	$2.10 \pm 0.11$	$2.83 \pm 0.22$	Binary regression (logit function)
Capillary suction	$1.48 \pm 0.84$	$3.97 \pm 0.98$	-
ETH method on laboratory specimens	$1.30 \pm 0.18$	$2.34 \pm 0.69$	Mean value from a fitted statistical distribution
ETH method on the structure	$0.68 \pm 0.72$	(Nonpickled: $1.13 \pm 0.59$ )	Mean value from a fitted statistical distribution
Cracked samples	0.2 to 1.2	1.8 to >5.8	The 5% fractile of 1.4003 is determined to 1.7% by cem.wt.

Note: If not stated otherwise, the 1.4003 is pickled. The results are graphically summarized in Figure 4.



**FIGURE 3** Measured critical chloride content ( $C_{crit}$ ) (dots) and fitted statistical distributions (lines) for pickled 12% chromium steel 1.4003 (red solid lines) and unpickled carbon steel 1.0439 (blue) in CEM I systems. Nonpickled 1.4003 is depicted with red dashed lines. (a) Lollipop experiments, (b) ECISS experiments (results from the binary regression), (c) potentiostatic electrochemical test according to DIN EN 480-14, (d) capillary suction test. Moderately accelerated ETH method on laboratory specimens (e) and on specimens from a bridge (nonpickled) (f) [Color figure can be viewed at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)]



**FIGURE 4** Summarized results for critical chloride content ( $C_{crit}$ ) with different methods. The bars depict the mean values, the standard deviation is indicated by the whiskers. \*Nonpickled 1.4003 (depicted with the shaded area) [Color figure can be viewed at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)]

mixed-in chloride concentrations (1.0%, 1.5%, 2.0% chloride by cem.wt., etc.). The result is the binary information; steel corrosion initiates (Yes/1) or steel remains inactive (No/0). As mentioned above, the statistical postprocessing of the ECISS data requires binary regression (here the logit model was used for regression analysis), see Figure 3b.

Because the data of this test is limited (in total 30 trials) and due to the variability of the results the confidence bound is relatively broad. Nevertheless, according to the ECISS test criteria, the  $C_{crit}$  is 2.5% chloride by cem.wt. and this value corresponds to the 10% probability of corrosion initiation. Whereas, based on the binary regression the mean value was 3.37% chloride by cem.wt. with a standard deviation of 0.42% chloride by cem.wt. Consequently, the ECISS test criteria correspond to a low corrosion probability with regard to the chloride threshold and lead to a great safety margin.

It should be mentioned that the ECISS test is not applicable to carbon steel, as explained in Reference [23]. The test procedure was found not to allow steel passivation in the alkaline mortar due to the immediate contact with chloride ions in combination with the anodic polarization. Consequently, carbon steel corrodes and during the visual examination of the surface, the appearance of oxide products leads to a binary test result “No/0”, even though the corrosion current density does not exceed the limiting value of 80 mA/m<sup>2</sup>.

### 3.3 | Potentiostatic electrochemical test according to DIN EN 480-14

On the basis of the criteria listed in Section 2.3, the chloride threshold value is the lowest chloride content

where corrosion was initiated during the *accelerated polarization test*. On the basis of the results shown in Figure 3c, the first corrosion initiation for series 1.4003 occurred at 2.3% chloride by cem.wt. For series 1.0439, the first corrosion initiation occurred at 1.5% chloride by cem.wt.

However, to ensure comparability with other methods, the mean value of the  $C_{crit}$  must be determined. As this test method only contains discrete values as results, these must be transformed into a binary data set consisting of 0 and 1. For this purpose, the result for the respective chloride content is evaluated as 1 for “yes corrosion” and 0 for “no corrosion.” As explained above, the logit model was used with a confidence interval of 95%. The graphical representation of the binary regression is shown in Figure 3c, the mean value for  $C_{crit}$  for series 1.4003 is 2.83% chloride by cem.wt. with a standard deviation of 0.22%. The mean value for series 1.0439 is 2.10% chloride by cem.wt. with a standard deviation of 0.11%.

### 3.4 | Capillary suction test

The determination of the chloride content using LIBS on a selected carbonated sample is shown as an example in Figure 5. Both iron and chloride ions were determined in this area. Areas with measurable iron content are not considered for the determination of the chloride content. According to Reference [37] up to 11.6 wt.% FeCl<sub>3</sub> is contained in the corrosion products. Values from these areas are not representative for the chloride content in the phase boundary range steel-mortar. In addition to the chloride and iron contents, the carbon content was also shown informatively. Subsequently, the mean value of the chloride content is determined for the POI.

Figure 5 shows the measuring range in the POI and the area for determining the chloride content within the POI, the hatched area is not taken into account for determining the chloride content because the chloride content within the corrosion products is not comparable with the one of the surrounding cementitious matrix.

When using the mat crosses (Figure 1d right sample), to which the welding wires from 1.4571 were attached by a TIG (tungsten inert gas welding) welding process, corrosion in the area of the TIG welding seam occurred in carbonated mortar on two of the six mat crosses. No significant chloride content could be analyzed in the area of the weld seam. That occurs due to a disturbed or not formatted chrome oxide layer in the heat-affected zone of the welding process, especially if the chrome content of the substrate is low.



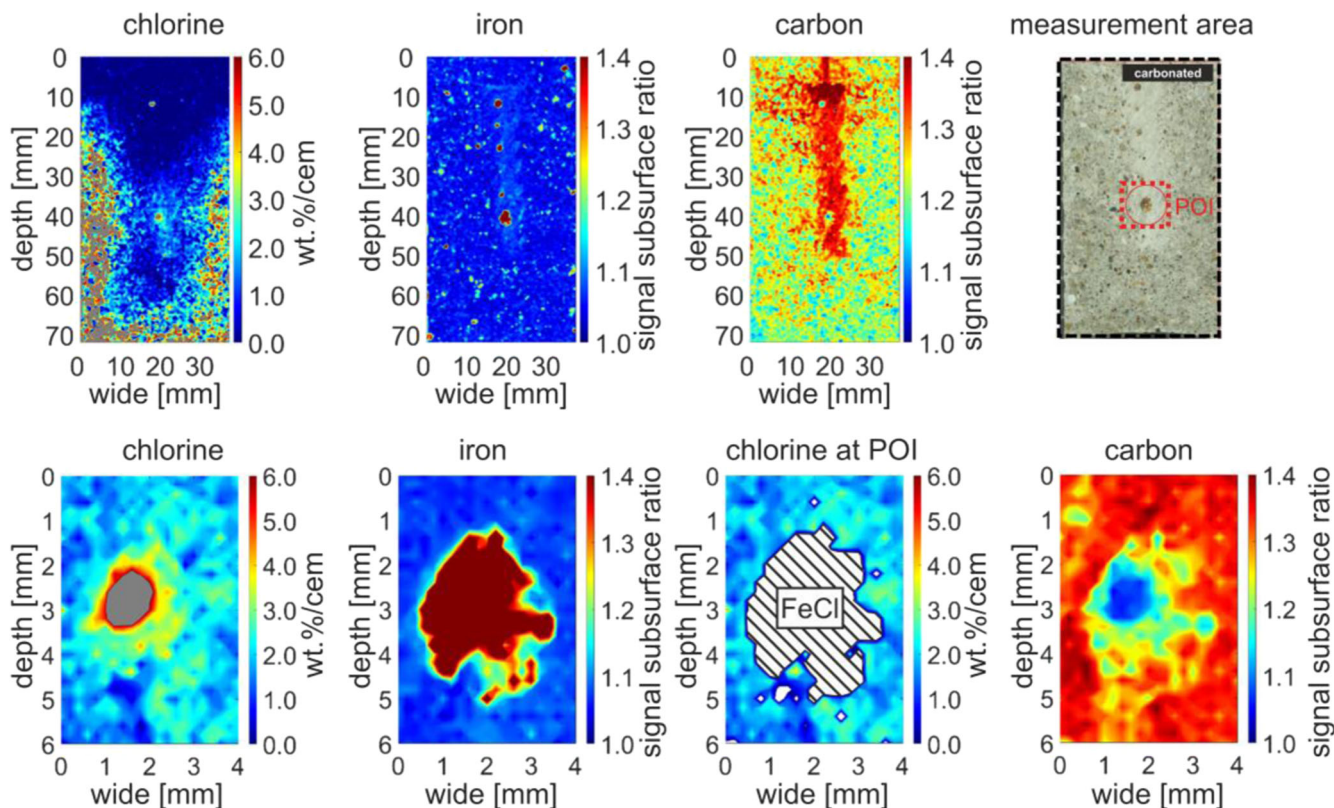


FIGURE 5 Graphical representation of the laser-induced breakdown spectroscopy measurement results on a representative carbonated sample and the point of interest [Color figure can be viewed at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)]

Figure 6 shows the distribution function of the normal distribution for both materials and for 1.4003 in alkaline and carbonated mortar. The following values are listed in Table 3. The mean value for the *suction test*, in the case of 1.4003 and alkaline mortar is 3.97% chloride by cem.wt. and for 1.0439 1.48% chloride by cem.wt. The standard deviation is also listed in Table 3. In the case of carbonated mortar, the values for 1.4003 are also given in Section 4.4.4.

### 3.5 | Moderately accelerated ETH-method

The results of  $C_{\text{crit}}$  determined with the specimens manufactured in the laboratory are given in Figure 3e. The mean values of  $C_{\text{crit}}$  equal 1.30% chloride by cem.wt. for nonpickled carbon steel (1.0439) and 2.34% chloride by cem.wt. for pickled steel 1.4003 (Table 3). The standard deviation of 1.4003 is larger than the one of 1.0439 (0.69 instead of 0.18).

The results of  $C_{\text{crit}}$  for the samples taken from a reinforced concrete structure are given in Figure 3f. Both steel grades show lower mean values of  $C_{\text{crit}}$  than in the laboratory specimens (Table 3). The standard deviation

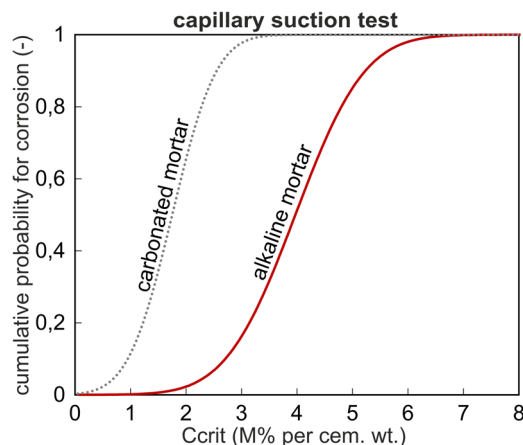


FIGURE 6 Critical chloride content ( $C_{\text{crit}}$ )-fitted statistical distributions for pickled steel 1.4003 in carbonated and alkaline mortars exposed to the capillary suction test [Color figure can be viewed at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)]

for both steels is similar. Note that both steels in the structure were nonpickled.

In Figure 3e,f, the experimental data was described by a lognormal distribution fit.



### 3.6 | Depassivation behavior in cracked specimens exposed to chloride ions

As the final results of this series of experiments will be published separately, the results are briefly summarized in Table 3, showing a significantly higher range of  $C_{\text{crit, crack}}$  for the 1.4003. The 5% fractile value of  $C_{\text{crit, crack}}$  is 1.7% chloride by cem.wt.

This high resistance to chlorides in the crack can be expected as the 1.4003 has the possibility to develop a stable passive layer in the initially chloride-free concrete before chlorides penetrate the crack area.

### 3.7 | Summary

All different methods can give results for the  $C_{\text{crit}}$  in form of a statistical distribution. As will be discussed later, the experimental conditions of testing  $C_{\text{crit}}$  differ strongly from each other. Nevertheless, the  $C_{\text{crit}}$  measured in these experiments for pickled 1.4003 in alkaline mortar/concrete was between 2.3% and 4.9% chloride by cem.wt., while for 1.0439 steel in as-received (nonpickled) condition in alkaline mortar/concrete,  $C_{\text{crit}}$  was observed in the range 0.7%–2.1% chloride by cem.wt. (compare Table 3 and Figure 3).

## 4 | DISCUSSION

### 4.1 | Comparison of different experimental setups

The presented  $C_{\text{crit}}$  test methods show great variety with regard to exposure condition, test procedure, evaluation criteria, specimen size, and the number of specimens. However, all test methods have in common that they accelerate the corrosion initiation in different ways.

To compare the different results of  $C_{\text{crit}}$  obtained with the present tests and in particular to assess their applicability to practice, the experimental methods are reviewed in the following sections (compare Table 4).

#### 4.1.1 | Anodic polarization

Corrosion of steel in concrete is an electrochemical process that can be triggered through external anodic polarization. Under natural conditions, the potential of the reinforcement can differ considerably from the potential in polarized laboratory tests, such as the *ECISS test* or the *potentiostatic polarization test*, considered in this study. The reason for applying anodic polarization of the working electrode in  $C_{\text{crit}}$

testing for alloyed steels is essentially to accelerate the test, because at potentials representative for potentials of steel in atmospherically exposed concrete structures, very high chloride concentrations may be needed to trigger corrosion for these steels.<sup>[38]</sup> From an electrochemical viewpoint, anodic polarization increases the probability to exceed the pitting potential, which provokes corrosion initiation at lower chloride concentrations compared to the unpolarized state.<sup>[38]</sup>

Thus, all methods involving anodic polarization of the steel electrode under test lead to conditions that lead to a larger offset with practice.<sup>[3]</sup>  $C_{\text{crit}}$  obtained at potentials anodic to the potentials expected for steel in concrete under the relevant exposure conditions may be considered conservative (too low).

#### 4.1.2 | Chloride addition

As reviewed in detail in Reference [3], there are various ways to introduce chlorides into the mortar/concrete for  $C_{\text{crit}}$  tests. A simple approach is to add chlorides during the preparation of the cementitious material (“mixed-in”). While this allows achieving relatively high chloride concentrations in a short time, the approach is not well related to practice, where chlorides generally penetrate into the hardened concrete from external sources. Thus, approaches involving bulk diffusion or combinations of capillary suction and diffusion are generally adopted and acceleration compared to real-life exposure conditions is essentially achieved by using both shallow cover depths and high chloride concentrations in the laboratory exposure tests. Note that shallow cover depths require either small aggregates in the concrete/mortar mix (to avoid problems related to concrete compaction in zones between the rebar and the formwork) or reducing the cover depth after casting by means of concrete cutting.

For cementitious systems exhibiting sufficient capillary porosity, an additional accelerating effect can be obtained from wetting/drying exposure.<sup>[39]</sup> Finally, chloride ingress can be enhanced by applying an electrical field (migration). It may be noted that capillary suction tests, especially protocols including pre-drying,<sup>[10,40]</sup> and electrical migration tests may under some conditions lead to fast ingress of chloride and risk for “overshooting”  $C_{\text{crit}}$ . It is thus important to apply these techniques carefully to avoid obtaining optimistic (too high)  $C_{\text{crit}}$  with respect to practical conditions.

Assessing the effect of these different chloride addition techniques is not straightforward. This is because the different techniques affect the corrosion behavior in a number of interrelated ways. For instance, mixed-in chlorides may enhance chloride binding, and at the same time also raise the pore solution pH and influence the porosity.<sup>[41]</sup> Long

TABLE 4 Comparison of experimental methods including an assessment with respect to the test parameters that render the results conservative (tendency for too low critical chloride content [ $C_{crit}$ ]) or optimistic (tendency for too high  $C_{crit}$ ) with respect to practical conditions, here assuming wetting/drying exposure

Experimental Setup	Advantages	Disadvantages	Factors that make $C_{crit}$ conservative ("too low")	Factors that make $C_{crit}$ optimistic ("too high")
Pitting resistance equivalent number	Extremely simple and fast; no laboratory bias	No $C_{crit}$ , thus no service life predictions possible Not related to steel in concrete, thus not applicable to practice Only applicable to stainless steels	-	-
Lollipop	Not polarized No mixed-in chlorides Comparison for different steel grades possible	Long duration of test for alloyed steels (up to 2.5 months)	Mortar specimen High-quality steel-concrete interface High moisture content (immersion)	Mortar specimens
ECISS	Relatively fast No chloride testing needed No chloride testing needed Clear criterion of termination	Not representative for site conditions (anodic polarization, mixed-in chlorides, ...)	Anodically polarized	Mortar specimens
Potentiostatic electrochemical test according to DIN EN 480-14	Relatively fast Reproducible Procedure and criterion of termination is standardized	No comparison to mild steel is possible Not representative for site-conditions (anodic polarization, mixed-in chlorides) Low number of specimens	Anodically polarized	Short duration (24 h) Mortar specimens High moisture content (immersion)
Capillary suction	Not polarized No mixed-in chlorides Comparison for different steel grades possible	Long duration of experiment for alloyed steels (up to 12 months) LIBS results for $C_{crit}$ are difficult to compare to $C_{crit}$ with acidic dissolution	Mortar specimens High-quality steel-concrete interface High moisture content (immersion) Locally measured $C_{crit}$	Mortar specimens High-quality steel-concrete interface High moisture content (immersion) Locally measured $C_{crit}$
ETH method on laboratory specimens	Concrete specimens Not polarized No mixed-in chlorides Comparison for different steel grades possible	Long duration of experiment for alloyed steels (up to 12 months)	High-quality steel-concrete interface High moisture content (immersion)	High-quality steel-concrete interface High moisture content (immersion)

TABLE 4 (Continued)

Experimental Setup	Advantages	Disadvantages	Factors that make $C_{crit}$ conservative (“too low”)	Factors that make $C_{crit}$ optimistic (“too high”)
ETH method on the structure	concrete specimens realistic steel-concrete interface not polarized no mixed-in chlorides comparison for different steel grades possible	Long duration of experiment for alloyed steels (up to 24 months)	Accelerated ingress of chlorides (migration)	High moisture content
Cracked samples	Concrete specimens Realistic steel-concrete interface Low, practical polarization (60 mV) No mixed-in chlorides Comparison to 1.0439 is possible	Long duration of the experiment (XY months) up to >24 months	Laboratory scale with perfect ITZ concrete-steel	Permanent chloride load; investigation on separating crack

exposures in laboratory conditions, in particular at shallow cover depths, can lead to leaching effects modifying the pore solution composition (e.g., pH decrease), and the adoption of electrical migration techniques may affect chloride binding<sup>[42]</sup> and modify the concrete pore solution through the movement and generation of other species (e.g., hydroxyl ions). It can be concluded that due to the complexity of the influences of the different chloride addition techniques on various properties relevant for corrosion (pore solution composition, concrete porosity, chloride binding, etc.) it is not straightforward to assess if a certain chloride addition technique leads to conservative or optimistic results with respect to engineering practice. Nevertheless, as a general rule, the more the degree of acceleration, the more is the obtained  $C_{crit}$  likely to deviate from practical conditions.

#### 4.1.3 | Concrete moisture conditions

The different techniques to introduce chlorides discussed above are closely related to the resulting concrete moisture conditions. Bulk diffusion and migration generally mean moisture conditions close to capillary saturation. Wetting/drying cycles, on the contrary, may lead to concrete internal moisture conditions below capillary saturation. The moisture state achieved at the steel surface depends on the cover depth, concrete moisture retention properties, the duration of the wetting and drying intervals, and the conditions during the drying stage.

Experiments have shown that the  $C_{crit}$  of continuously wet exposed specimens is higher than values found for specimens exposed to wetting-drying cycles.<sup>[39]</sup> This is in accordance with results from Reference [43], showing that with long submerged exposure, high  $C_{crit}$  is to be expected. The influence of moisture on  $C_{crit}$  is also discussed in Reference [11] showing that more research needs to be conducted to understand the related effects in detail.

When it comes to assessing the effect of test conditions on the test result and its applicability to engineering practice, it should be noted that in marine and road-salt exposure, structures are often exposed to wetting-drying cycles, and only submerged structural members are continuously wet. The results from experiments in continuous wet exposure might therefore be too optimistic ( $C_{crit}$  too high) compared to cyclic conditions.

Finally, it has to be considered that chloride ingress in concrete is rarely homogeneous due to variability in the microstructure of the concrete<sup>[44]</sup> and potentially microclimates at the exposed surface. This variability in structures can hardly be represented in laboratory tests, simply due to the tendency for relatively homogeneous concrete within small laboratory samples compared to field concrete.

In summary, chloride ingress by means of bulk diffusion and capillary suction is considered the most practice-related condition but may lead to very long testing times, in particular for stainless steels that exhibit high  $C_{\text{crit}}$ . All methods used here involve immersion of the specimen in a test solution and thus lead to comparatively high moisture states. Thus, the obtained  $C_{\text{crit}}$  are generally optimistic with respect to road salt exposure including wetting/drying.

#### 4.1.4 | Laboratory-produced specimens/mortar

In a recent review, it was shown that the steel–concrete interface (SCI) is very inhomogeneous<sup>[45]</sup> and that different characteristics present at the SCI influence strongly the corrosion initiation of steel in concrete.<sup>[11]</sup> It was also shown that laboratory-produced specimens (especially, when mortar instead of concrete is used) are hardly representative for site conditions, in particular, that laboratory samples tend to show a more homogeneous SCI compared to reinforced concrete structures. This homogeneity at the SCI can lead to higher  $C_{\text{crit}}$  for laboratory samples than for structures, even when produced from the same materials (e.g. cement type and steel type). This means, that the results from laboratory samples might lead to more optimistic results (i.e., higher  $C_{\text{crit}}$ ) than what would be the case in reinforced concrete structures. For these reasons, we consider the test methods that employ concrete closer to the condition in engineering structures than methods based on mortar samples. Here, among the studied methods, only the ETH method employed concrete.

#### 4.1.5 | Evaluation criteria

All  $C_{\text{crit}}$  test methods require criteria to decide whether corrosion initiation took place or not. Here, the studied test methods employ either an electrochemical criterion or rely on visual examination of the steel surface after removal of the surrounding concrete.

For the tests with free rebar potentials (not externally polarized), such as the *lollipop test*, the *capillary suction test*, and the *ETH method*, a pronounced drop in potential was considered to indicate corrosion initiation. The electrochemical criterion was a sharp potential decrease,  $\Delta E$ , in the negative (cathodic) direction ( $\Delta E$  100–300 mV). However, since the OCP is a dynamic parameter and processes such as repassivation can lead to an increase of the potential after the drop,<sup>[46]</sup> the *ETH method* additionally required that the potential remained on the level after the drop for a certain period (here 7 days) before ending the test. Due to the comparatively long testing times of this method, the

chloride concentration is expected to increase only marginally during these 7 days. This additional criterion is based on Reference [10]. It is expected that the criteria used for the *lollipop* and the *capillary suction tests* led to a more conservative  $C_{\text{crit}}$  compared to the *ETH method* because the *ETH method* requires a more stable corrosion initiation process to occur than the other two methods.

The  $C_{\text{crit}}$  tests with potentiostatic polarization use an increase in corrosion current density as an electrochemical criterion. This criterion is less prone to interpretation difficulties than those based on OCP monitoring, but, as discussed above, the potentiostatic control leads to fewer practice-related conditions during the test.

#### 4.1.6 | Synthesis

On the basis of the discussion in the previous sections, the *ECISS test* and the *DIN EN 480-14 protocol* are considered the least practice-related tests, while the *lollipop test*, the *capillary suction test*, and the *ETH method* are considered to better represent the conditions expected in practice.

Nevertheless, all test methods studied in this study do not reflect practical conditions. The  $C_{\text{crit}}$  results have, therefore, always to be judged according to the used experimental conditions and to the planned exposure in practice.

## 4.2 | $C_{\text{crit}}$ values

Despite the very different testing conditions, all different experimental test methods studied in this study indicate that  $C_{\text{crit}}$  of steel grade 1.4003 (pickled) is between 2.3% and 4.9% chloride by cement weight. As is apparent from Figure 4, both the average values and the standard deviations obtained from the series tested with the various experimental setups, are very different.

As the differences between the experimental setups are large and cover situations that may be considered optimistic as well as conservative with respect to site conditions, we expect that  $C_{\text{crit}}$  in practice will fall in the range observed here. In other words, the value of  $C_{\text{crit}}$  of steel grade 1.4003 (pickled) will most probably only rarely be lower than the 2.3% chloride by cem.wt. This result is consistent with  $C_{\text{crit}}$  of other alloyed steels.<sup>[23]</sup> The observed  $C_{\text{crit}}$  of steel grade 1.0439 was in the range of 1.2%–1.5% chloride by cem.wt.

These results obtained for mild carbon steel may be considered relatively high when compared to experiences from engineering practice,<sup>[47]</sup> as is also visible from Figure 4. This discrepancy can be explained by the differences in steel–concrete interfacial conditions (e.g., heterogeneous concrete in a structure compared to relatively

homogeneous mortar in laboratory samples). Another possible explanation for the discrepancy between relatively high  $C_{\text{crit}}$  in laboratory testing compared with experience from practice may be the size effect,<sup>[48–50]</sup> that is, the fact that all tested laboratory specimens were relatively small (only a few centimeters in length). It should be noted that the size effect similarly applies to mild carbon steel<sup>[48–50]</sup> as to stainless steels.<sup>[51]</sup> This needs to be borne in mind when translating values determined on small-scale specimens to engineering conditions, which, however, is beyond the scope of this paper. For further details, the reader is referred to Reference [52].

### 4.3 | Comparison of the two tested steel types

For comparison of the corrosion susceptibility of different steels, sometimes a ratio between the  $C_{\text{crit}}$  of 1.4003 and 1.0439 is reported. This approach may be suitable when testing different steel grades with a given test method in laboratory environments and to allow for a ranking of the different steel grades in terms of their corrosion resistance. Note, however, that rankings are only applicable to the conditions under which they were determined and cannot necessarily be translated to other conditions. It was also apparent from this study that the ratio between  $C_{\text{crit}}$  of steel types 1.4003 and 1.0439 was different between the different test methods. This suggests that the ratio is dependent on the test method and the corresponding test conditions.

Thus, caution should be exercised when using the approach of a ratio of  $C_{\text{crit}}$  of different steel grades for the purpose of translating test results to engineering practice. In particular, we do not recommend using the ratio between laboratory  $C_{\text{crit}}$  values of alloyed steel and carbon steel to infer  $C_{\text{crit}}$  values for the alloyed steel under practical conditions, simply by multiplying a commonly reported carbon steel threshold value such as 0.4% chloride by cem.wt. with this factor. In addition to the limited applicability of rankings to other conditions mentioned above, another difficulty is that carbon steel is not a well-defined standard. This is apparent from the extremely large scatter reported for  $C_{\text{crit}}$  of carbon steel in literature,<sup>[3,53]</sup> where, for mild carbon steel in Portland cement systems a range of values from virtually 0 to almost 4% chloride by cem.wt. can be found.

## 4.4 | Further influencing parameters

### 4.4.1 | Cracks

A brief presentation of investigations on the corrosion behavior in cracked concrete specimens can be found

in Section 3.6. The test results show that the samples reinforced with 1.4003 only permanently depassivate at significantly higher chloride contents than the test specimens reinforced with conventional reinforced steel (1.0439).

### 4.4.2 | Steel surface condition

One of the most important influence parameters on the corrosion resistance of 1.4003 is its surface condition. Pickled 1.4003 showed a higher  $C_{\text{crit}}$  than unpickled 1.4003, by up to a factor of 2. This finding is in agreement with other studies, where it was shown that removing rust and mill scales from rebars, for example, by sandblasting, polishing, or pickling, significantly improved the resistance against corrosion.<sup>[54–57]</sup> In a recent literature review,<sup>[11]</sup> the surface condition of the steel was revealed to be one of the most important influencing factors for  $C_{\text{crit}}$  of carbon steel in concrete. In fact, removing these scales on carbon steels can raise  $C_{\text{crit}}$  by a factor of up to 2–3.

As the application of pickled steel 1.4003 (brand “Top12” as marketed by Steeltec Group) in practice started around the year 2016, most of the long-term experiences in practice are so far with unpickled 1.4003. It is expected that the corrosion resistance of the structures built after 2016 is higher than the unpickled 1.4003 from older structures.

### 4.4.3 | Carbonated concrete

In the case of carbonated concrete,  $C_{\text{crit}}$  of 1.4003 was found to decrease drastically. This is based on the influence of the  $\text{Cl}^-/\text{OH}^-$  ratio in the pore solution.<sup>[11]</sup> Figure 6 shows the cumulative frequency of the capillary suction test with carbonated specimens. The mean value for  $C_{\text{crit}}$  at carbonated CEM I-based specimen with a pH value less than 9 was 1.75% chloride by cem.wt. and the standard deviation was 0.63.

### 4.4.4 | Welded areas

Welded areas at 1.4003, which are welded by TIG or other welding processes, with a high amount of energy and heat at a local area reduces the already low chromium content for the passive layer formation by the formation of chromium carbonates. As a result of this, the formation of corrosion products could be observed at welded chloride-free regions in carbonated mortars. In those areas, the 1.4003 does not have higher corrosion resistance, compared to 1.0439, anymore.



## 5 | CONCLUSIONS

To choose the needed steel grade to ensure the service life for a given application, such as reinforced concrete structures in the chloride-contaminated environment, test methods, predictive models, or other approaches (e.g., PREN) are needed. From the experiments performed in this study, with a number of selected test methods, we draw the following major conclusions:

- The conditions for test methods to determine  $C_{\text{crit}}$  have a major influence on the test results. Thus, if different steel grades are to be compared, they should be tested with one common test method. Results from different methods can differ by a factor of two, even if they are based on similar principles. This has a considerable influence on service life predictions/calculations
- Some of the studied methods are only applicable to stainless steel (*ECISS*, *potentiostatic test*) but cannot deliver results for carbon steel.
- Under the conditions of the tests performed in this study  $C_{\text{crit}}$  of steel grade 1.4003 (pickled) is between 2.3% and 4.9% chloride by cement weight. The observed  $C_{\text{crit}}$  of steel grade 1.0439 was in the range of 1.2%–1.5% chloride by cem.wt. Note that for conditions different from the ones studied in this study (other binder systems, other moisture conditions, other steel surface treatment)  $C_{\text{crit}}$  may deviate from the ranges indicated above.

On the basis of theoretical considerations and literature review, we make the following recommendations:

- All test methods employ conditions modified with respect to engineering practice. Generally, methods with a higher degree of such modifications can yield results faster, but this gain in experimental time may be at the expense of the applicability of the results to practice. Here, this applies in particular to the *ECISS test* and the *potentiostatic polarization test*. Other tests, such as the *lollipop test*, the *capillary suction test*, and the *ETH method*, are believed to include fewer modifications with respect to practical conditions and thus the results are considered more practice-related. Similarly, regarding the heterogeneity of the steel-concrete interface and its effect on  $C_{\text{crit}}$ , we recommend that specimens made from concrete (not mortar or cement paste) are used.
- If  $C_{\text{crit}}$  is to be used in service life modeling, we thus recommend adopting methods, such as the *lollipop test*, the *capillary suction test*, and the *ETH method*. The results from the *ETH method* may be considered as the closest to engineering practice and may thus, in the absence of long-term experience from practice, be used as an estimate of  $C_{\text{crit}}$ . For probabilistic modeling,  $C_{\text{crit}}$  for steel type

1.4003 may be described with a lognormal distribution with a mean value and standard deviation of 2.3% chloride by cem.wt. and 0.69% chloride by cem.wt., respectively (which corresponds to  $\lambda = \mu_{\ln x} = 0.81$  and  $\epsilon = \sigma_{\ln x} = 0.29$ ). Nevertheless, caution should still be exercised when using the test results because of the various factors that may render the  $C_{\text{crit}}$  optimistically high with respect to practice as discussed in this paper (steel-concrete interface quality, high moisture content, small specimen size).

- If the primary objective is a ranking of different steel grades, fast methods, such as the PREN, the *ECISS test*, and the *potentiostatic polarization test*, may be suitable approaches. Note, however, that rankings are only applicable to the conditions under which they were determined and cannot necessarily be translated to other conditions.

## ACKNOWLEDGMENT

The authors acknowledge the financial support by Steeltec Group for funding these research projects.

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**How to cite this article:** C. Boschmann Käthler, G. Ebell, S. Keßler, Y. Schiegg, C. Dauberschmidt, U. M. Angst. A comparison of methods to assess the resistance of reinforcing steel against chloride-induced corrosion in concrete—Particular consideration of 12% chromium steel. *Mater. Corros.* **2022**;73:306–325. <https://doi.org/10.1002/maco.202112826>