# Sodium Sulfate Reactivity Test of Carbon Anodes in Aluminium Production

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**Abstract**

Prebaked carbon anode is the primary consumable material in the aluminum reduction cell. Its quality directly affects the durability of carbon anodes and the costs associated with the Hall-Héroult aluminum production process. The conditions of traditional anode quality control tests, such as $CO_2$ and air reactivity tests, differ from those of industrial electrolytic cells. This study presents an alternative Sodium Sulfate Reactivity (SSR) test, which could better simulate real electrolysis conditions by incorporating in-situ $CO_2$ generation and liquid-solid interactions in the molten sodium sulfate bath. An experimental setup was used to immerse carbon anode samples in a molten sodium sulfate bath. Initial tests showed the importance of optimizing test parameters, particularly test temperature and time, to achieve measurable mass loss results. At a test temperature of 990 °C and a test time of 30 min, measurable mass losses were obtained for samples extracted from industrial baked anodes. Multiple linear regression analysis showed that both centered apparent density ($p = 0.0007$) and anode-to-anode variability ($p = 0.0387$) have a significant correlation to the mass loss, with a global $R^2$ of 0.870.

**Keywords:** Aluminium electrolysis, Carbon anode, Carbon consumption, $CO_2$ reactivity, Sodium sulphate reactivity.

## 1. Introduction

Aluminium is produced from its alumina ($Al_2O_3$) ore through the Hall-Héroult process. In this process, $Al_2O_3$ is reduced in the electrolytic cell. The electrolytic cell is composed of a carbon cathode, carbon anodes, and molten cryolite ($Na_3AlF_6$) as electrolyte. The cells typically operate at 940–980 °C [1]. The prebaked carbon anode is the primary consumable material in the reduction cell, which, in addition to conducting electric current, acts as the reducing agent in the electrochemical reaction:

$$2Al_2O_3 + 3C \rightarrow 4Al + 3CO_2 \quad (1)$$

Based on this reaction, the theoretical consumption of anodes is 333 kg per ton of aluminium produced. However, additional consumption is observed due to side reactions such as air oxidation, the Boudouard reaction, and preferential binder oxidation (dusting) [2–4]. Excessive carbon consumption increases Al production costs and $CO_2$ emissions [4, 5]. This excessive carbon consumption is influenced by a number of material and processing factors. Processing parameters, i.e., mixing and compaction of anode paste and baking temperature (BT), and anode

properties, i.e., baked density, pore size distribution, and chemical composition, may affect anode reactivity [6].

Carbon anode reactivity is conventionally assessed through ISO standard tests. In these tests, the carbon anode is exposed to the flow of reactive gases ($CO_2$ or air) in a furnace, and the mass loss of the samples is measured [7, 8]. However, the conditions of these indirect methods differ from those of real electrolysis conditions, limiting their ability to fully represent industrial anode performance in the reduction cell. Specifically, these tests do not consider the effect of molten electrolyte environment, in-situ $CO_2$ generation, or temperature gradients present in operational reduction cells.

Given these limitations, we propose revisiting a complementary tool to assess anode reactivity under conditions closer to those of real electrolysis. This approach is based on sodium sulfate reactivity (SSR), and we believe that it will potentially enable the refinement of the anode reactivity tests and provide a detailed understanding of the effect of anode properties on carbon anode reactivity and consumption in the cell.

The SSR test was used in the industry between the 1950s and 1990s to evaluate anode reactivity. This test assesses the reactivity of an anode sample immersed in molten sodium sulfate. Therefore, it provides a reactive liquid environment and, consequently, liquid-solid interaction. At temperatures above 884 °C and in the presence of carbon, $Na_2SO_4$ is reduced to $Na_2S$, and carbon is oxidized to CO and $CO_2$. According to the literature, the following chemical reactions are possible in in this system [9, 10]:

$$Na_2SO_{4(l)} + 4C_{(s)} \rightarrow Na_2S_{(l)} + 4CO_{(g)} \quad (2)$$

$$2Na_2SO_{4(s\ or\ l)} + C_{(s)} \rightarrow 2Na_2O_{(l)} + 2SO_{2(g)} + CO_{2(g)} \quad (3)$$

$$C_{(s)} + CO_{2(g)} \rightarrow 2CO_{(g)} \quad (4)$$

$$Na_2SO_{4(s\ or\ l)} + CO_{(g)} \rightarrow Na_2O_{(l)} + SO_{2(g)} + CO_{2(g)} \quad (5)$$

$$3Na_2S_{(l)} + Na_2SO_{4(l)} \rightarrow 4Na_2O_{(l)} + 2S_{2(g)} \quad (6)$$

$$Na_2S_{(s)} + 2CO_{2(g)} \rightarrow Na_2CO_{3(l)} + COS_{(g)} \quad (7)$$

$$Na_2CO_{3(l)} + CO_{(g)} \rightarrow 2Na_{(g)} + 2CO_{2(g)} \quad (8)$$

$$Na_2SO_{4(l)} + 2C_{(s)} \rightarrow Na_2S_{(l)} + 2CO_{2(g)} \quad (9)$$

Reactions (3) and (9) generate $CO_2$ directly, whereas reaction (2) generates CO, which can be transformed to $CO_2$ via the Boudouard reaction (4). $CO_2$ can also be generated via secondary reactions (5) or (8). Although the specific contribution of each reaction is undetermined, several in-situ $CO_2$ generation mechanisms are identified. The generated $CO_2$ can subsequently react with the carbon anode at the solid/liquid interphase, similar to the carbon/cryolite system.

Anode structural characteristics, including porosity and apparent density, could influence mass transport of $CO_2$ from the solid/liquid interface into the anode and consequently affect the mass

loss in the SSR test. This can provide critical insight for optimizing the anode manufacturing process. In one document published by Wilkening [11] in 1994, this test was used to reveal its sensitivity to the anode baking temperature. Wilkening used an experimental setup to conduct the SSR test in which the carbon anode, connected to a thermocouple, was placed inside the molten sodium sulfate in a vertical furnace at 1 000 °C [11]. After 30 min, the sample was withdrawn and washed using an extractor. They showed that the SSR test has a greater sensitivity to the baking temperature (BT) of the anode compared to other reactivity tests using air or $CO_2$ in the gas phase. For instance, up to a BT of approximately 1 050 °C, air reactivity shows a slight variation, suggesting that it is independent of the BT [11]. However, SSR test shows great sensitivity to the BT up to 1 200 °C. In the current work, we revisited the SSR method, with particular focus on refining the test parameters and exploring correlations of the SSR reactivity with anode properties.

## 2. SSR Method

### 2.1 Immersion of the Sample in the Bath

The experimental setup used to perform the SSR test consists of a vertical crucible furnace (Lindberg MPH, USA, Model 56611) and a 280 mL (Ø88 × 72 mm) porcelain crucible filled with 230 g of $Na_2SO_4$ (Anhydrous, 99.99 %, Sigma-Aldrich) placed in a stainless-steel crucible of (Ø98 × 100 mm) with a handling rod. The assembly (Figure 1) is lowered into the vertical furnace, and the $Na_2SO_4$ is melted. During the test, the temperature of the melt is monitored by an alumina-sheathed, type-K thermocouple inside the porcelain crucible. When the temperature reaches the initial temperature (between 1 000 °C and 1 020 °C), the carbon sample is immersed in the melt through a hole in the insulator cover. The carbon sample is a cylindrical core of a baked anode, screwed to a rod, allowing to maintain it at the bottom of the porcelain crucible.

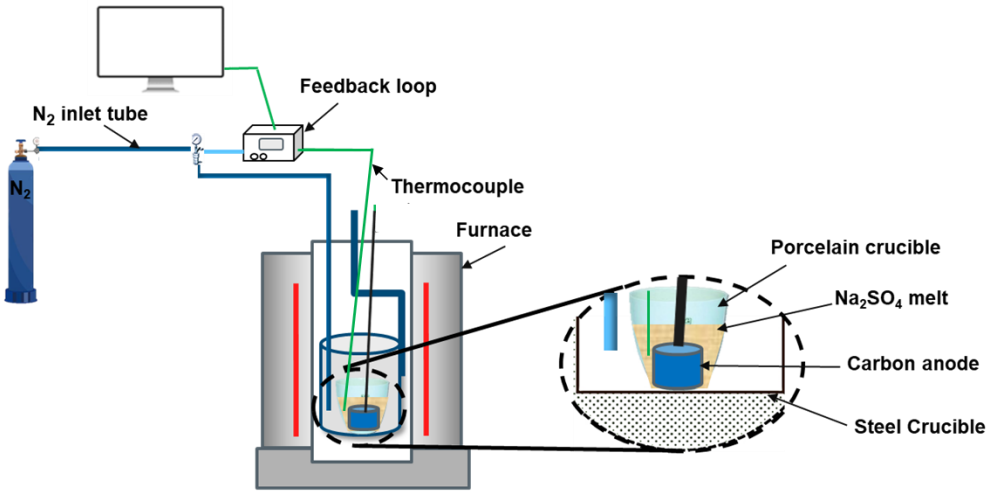


**Figure 1. Experimental arrangement for sodium sulfate test.**

When the anode sample is immersed in the melt, its temperature first decreases to a minimum temperature and then starts to increase. After reaching a constant test temperature ($T_{test}$) of 950–1 000 °C, the specimen is left in the $Na_2SO_4$ bath for the specified test time. The rise in the melt temperature is suppressed by blowing $N_2$ into the space between the porcelain and the stainless-steel crucible via two stainless-steel tubes. $N_2$ blowing is regulated by a feedback loop comprising a temperature controller (Love 16B-23) and a solenoid valve (ASCO, Model WWG4ELC1) connected to the thermocouple inside the melt, and allows controlling the melt temperature to ± 1 °C. At the end of the test time, the sample is withdrawn from the melt. The stainless-steel

crucible and the porcelain crucible are also withdrawn from the tubular furnace, and the melt is poured into a steel pan for further characterization.

### 2.2 Post-cleaning and Analysis

The salt residue, solidified on the sample surface, is extracted with water using a Soxhlet extraction apparatus, ensuring precise mass loss measurements (Figure 2). Then the sample is dried for 24 h at 100 °C. The mass loss percentage is calculated using the following equation.

$$Mass\ loss = \frac{m_i - m_f}{m_i} \times 100 \quad (10)$$

Where $m_i$ and $m_f$ are the initial and final sample masses, respectively.

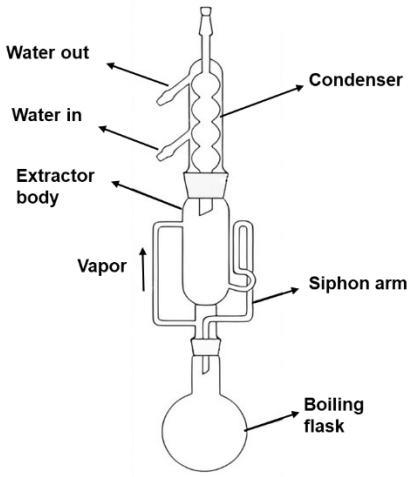


**Figure 2. Schematic of Soxhlet apparatus. Adapted from Ref [12].**

## 3. Evaluation and Optimization of SSR Test Parameters

The objective of this test series was to define a sensible range for operating parameters. More specifically, we varied temperature and test time in order to obtain measurable and repeatable mass loss results.

### 3.1 Initial Tests with Lab-scale Anodes

The most important test parameter that needs to be defined is the $T_{\text{test}}$. If the $T_{\text{test}}$ is too low, it can result in low mass loss and reduced sensitivity to detect meaningful differences between samples. Conversely, if the $T_{\text{test}}$ is too high, the residual sample mass may be too low, or it could be completely consumed regardless of sample differences. The test time is also an important parameter, and too short or too long test times could have effects similar to those of the $T_{test}$. Therefore, both $T_{\text{test}}$ and test time must be carefully adjusted to ensure that mass loss remains within a measurable and differentiable range.

The initial SSR tests were conducted using the available six lab-scale anode samples (samples Lab1 to Lab6 in Table 2). The baked apparent densities of these samples ranged between 1.45 g/cm$^3$ and 1.54 g/cm$^3$. More details on the production of lab-scale anodes are given in reference [13]. The diameter of the anode samples was reduced to 38 mm using a lathe, and they were cut to 25 mm lengths using a bandsaw. For these first tests, the furnace was preheated to 1 000 °C, and the $T_{\text{test}}$ was set to 950 °C, knowing that it will decrease upon introduction of the sample. Test times of 10 min, 20 min, and 30 min were chosen.

As summarized in Table 2, the results indicated very low mass loss (< 4 %) for the samples tested at a test time of 10 min and 20 min (Lab1–Lab4). For the tests made with a test time of 30 min (Lab5 and Lab6), there was a variation in mass losses, with values of 14.14 % and 4.21 %, respectively. While the variation between those two results suggests a degree of sensitivity, the mass loss of the Lab6 sample is still too low, which indicates that the selected test conditions were suboptimal for obtaining reliable reactivity measurements.

**Table 2. Summary of $Na_2SO_4$ results and the SSR test conditions using lab-scale anodes.**

| No. | Apparent density (g/cm$^3$) | Initial temperature (°C) | $T_{min}$ (°C) | Time to reach $T_{test}$ (min) | $T_{test}$ (°C) | Test time (min) | Mass loss (%) |
|---|---|---|---|---|---|---|---|
| Lab1 | 1.455 | 1 000 | 924 | 2 | 950 | 10 | 3.85 |
| Lab2 | 1.508 | 1 000 | 896 | 5 | 950 | 20 | 2.40 |
| Lab3 | 1.541 | 1 000 | 906 | 5 | 950 | 20 | 3.76 |
| Lab4 | 1.505 | 1 000 | 925 | 5 | 950 | 10 | 3.07 |
| Lab5 | 1.505 | 1 000 | 917 | 4 | 950 | 30 | 14.14 |
| Lab6 | 1.531 | 1 000 | 904 | 6 | 950 | 30 | 4.21 |

To maximize sensitivity and reduce other unwanted sources of error, the test conditions must be controlled well as much as possible. Figure 3 shows a representative temperature profile recorded by the thermocouple inside the bath for sample A1 (see Figure 4). A significant temperature drop (from 1 020 °C to approximately 920 °C) occurred immediately after sample introduction, followed by a delay before the $T_{test}$ was reached. This delay introduced a reaction period while the temperature is not under control, and it probably caused some consumption of the anode, even at a temperature lower than the $T_{test}$.

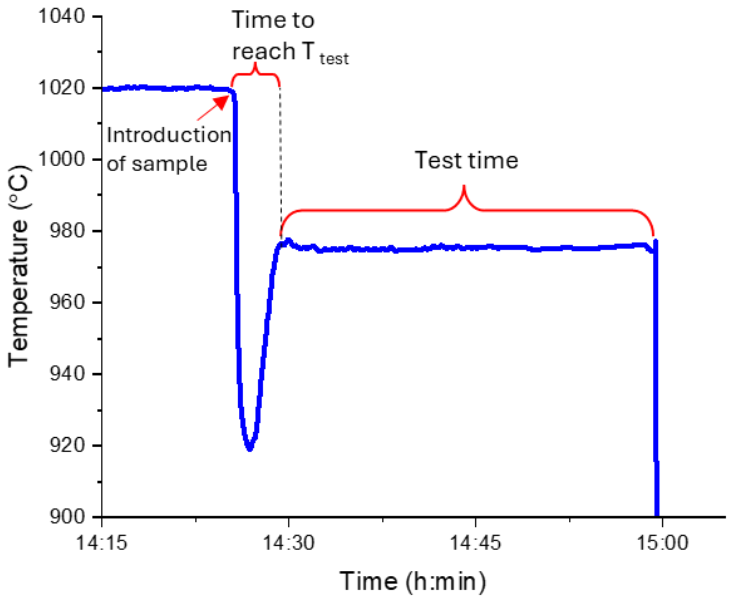


**Figure 3. Temperature of bath vs. time (sample A1).**

In some cases, partial solidification of the melt was also observed, which temporarily prevented contact between the sample and the liquid phase. Although this temperature remained above the melting point of $Na_2SO_4$ (884 °C), it approached the threshold where the melt can begin to partially solidify, particularly near cooler surfaces or due to local thermal gradients. These factors

introduced uncertainty in the real test conditions experienced by the sample and contributed to the observed variability in mass loss.

Based on these observations, the following improvement was introduced in the subsequent tests to address the undesirable thermal effects and increase the reproducibility. To reduce heat loss following sample introduction, the top of the furnace was insulated as effectively as possible. Due to the need to insert $N_2$ tubes, a thermocouple, and the sample handling rod, a fixed insulated cover could not be used. Instead, high-temperature insulating wool was used. The effectiveness of this insulation depends on both the thickness and placement of the wool, as well as the speed at which it was repositioned after the sample was introduced. After the first iterations, the method to insulate the cover was more efficient and consistent.

### 3.2 SSR Test Refinement

The objective of the second experimental series was to refine the SSR test parameters to achieve more consistent and representative mass losses in the target range of 20 % to 50 %, while minimizing variability introduced by other uncontrolled factors. Lab-scale anodes were not ideal for this purpose due to limited availability and significant variations in baked apparent density. The test samples must be as uniform as possible to study the effect of test parameters.

To reach such uniformity, cores were extracted from two industrial prebaked anode blocks provided by Alcoa Corporation (Figure 4). This approach enabled the preparation of a larger number of test specimens. These larger core samples (Ø50 × 400 mm) were machined to Ø38 mm using a lathe and then cut into 25 mm lengths. The resulting specimens (Ø38 × 25 mm) were sequentially numbered as identified in Figure 4.

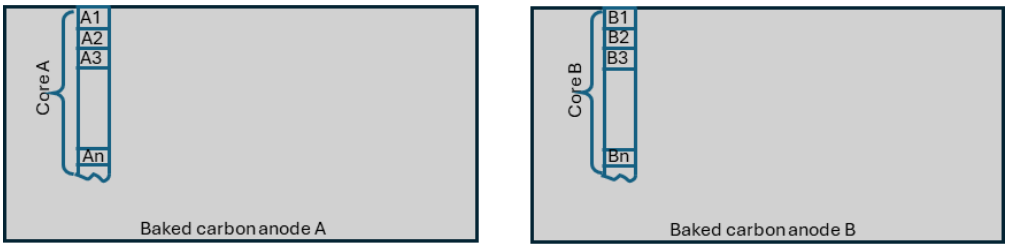


**Figure 4. Position of cores and numbering of the samples based on their position in the large cores of the baked carbon anodes.**

The results of SSR test using the extracted industrial samples are summarized in Table 2. For confidentiality reasons, each apparent density value was mean-centered, and the individual variations from the mean were reported. To minimize the temperature drop upon sample immersion and the time to reach the $T_{test}$, the initial temperature of the sodium sulfate melt was increased to the maximum achievable temperature for the setup.

A test time of 30 min was selected so that the time required to reach $T_{test}$ would be short compared to the total test time, thus reducing its impact on the measured mass loss. According to the results from the first series (Lab1 to Lab6 in Table 1), 950 °C was probably too low to induce sufficient reactivity. As a result, higher $T_{test}$ of 965 °C, 975 °C, and 990 °C were selected for evaluation. The single test performed at 965 °C (sample A8) resulted in a low mass loss of 1.37 %. Tests conducted at 975 °C (samples A1, A6, and A7) produced variable results, with mass losses ranging from 3.70 % to 22.79 %. However, the results obtained with a $T_{test}$ of 990 °C were more consistent and showed significant mass loss values. Samples A9, A10, and A11 were chosen for this test since they had very similar apparent density. These samples showed mass losses of

23.7 %, 23.6 %, and 26.2 %, respectively, which demonstrate that this temperature provided more stable and reliable conditions for SSR evaluation.

**Table 2. Summary of the SSR test results and conditions using industrial anodes.**

| No. | Centered apparent density (g/cm³) | Initial temperature (°C) | $T_{min}$ (°C) | Time to reach $T_{test}$ (min) | $T_{test}$ (°C) | Test time (min) | Mass loss (%) |
|---|---|---|---|---|---|---|---|
| A8 | 0.023 | 1 010 | 914 | 5 | 965 | 30 | 1.370 |
| A1 | -0.031 | 1 020 | 919 | 4 | 975 | 30 | 19.91 |
| A6 | -0.009 | 1 020 | 907 | 6 | 975 | 30 | 3.700 |
| A7 | -0.001 | 1 020 | 913 | 4 | 975 | 30 | 22.79 |
| A9 | 0.020 | 1 015 | 914 | 7 | 990 | 30 | 23.72 |
| A10 | 0.020 | 1 015 | 901 | 7 | 990 | 30 | 23.58 |
| A11 | 0.014 | 1 010 | 912 | 10 | 990 | 30 | 26.24 |
| B3 | -0.071 | 1 000 | 923 | 9 | 990 | 30 | 45.90 |
| B2 | -0.046 | 1 020 | 917 | 5 | 990 | 30 | 31.38 |
| B1 | -0.046 | 1 020 | 910 | 4 | 990 | 30 | 27.63 |
| B12 | 0.018 | 1 020 | 980 | 4 | 990 | 30 | 16.19 |
| B8 | 0.037 | 1 020 | 907 | 9 | 990 | 30 | 15.55 |
| B7 | 0.023 | 1 020 | 907 | 8 | 990 | 30 | 10.00 |

## 4. Analysis of Variance (ANOVA)

With the test conditions established ($T_{test}$ of 990 °C, test time of 30 min), more tests were conducted using these conditions, with anode core B. Samples A9, A10, A11, and selected samples from core B were tested under the same conditions, but there was a variation in mass loss between 10.00 % and 45.90 %. To try to explain this variation, an analysis of variance (ANOVA) was performed, using a linear model, with the centered apparent density and origin of the industrial core (core A vs core B) as indicators. The centered apparent density of samples extracted from two large cores (core A and core B) is shown in Figures 5(a) and (b), respectively. The variation in centered apparent density across both cores was relatively small, remaining within ± 0.07 g/cm³. Figure 5(c) shows the mass loss results as a function of centered apparent density, where a different color was given for each original anode.

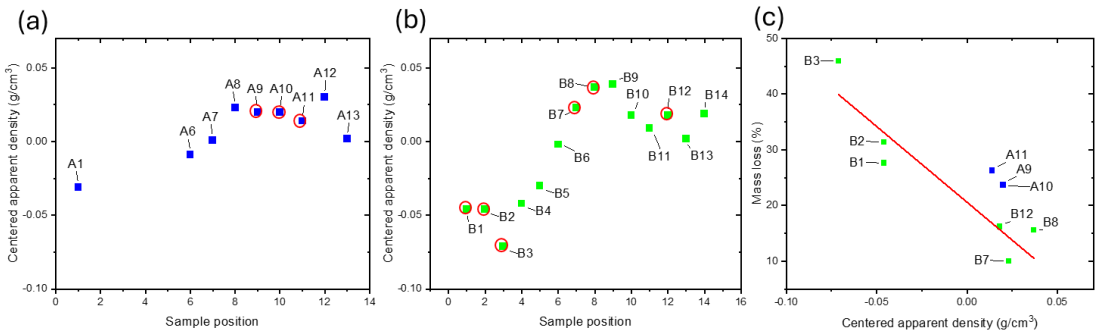


**Figure 5. (a) and (b) Apparent density of carbon anode samples as a function of sample position for the cores A and B, respectively (Samples with red circles were used for measuring the SSR test at $T_{test}$ of 990 °C). (c) Mass loss vs. apparent density at 990 °C for the sample of cores A and B with red circles. The red line fitted to the mass loss of the samples of core B.**

The analysis revealed that both parameters had a significant correlation. Centered density was the most important parameter, with a p-value of 0.0007, and the origin of the core had a p-value of

0.0387. $R^2$ value for the model was 0.870, and the root mean square error was 4.37 % mass loss. The regression line of mass loss as a function of density for anode B was added to Figure 5(c). One possible explanation for the correlation with density is that an increase in apparent density results in a more compact anode structure and causes a decrease in porosity and interparticle voids. It would correspond to reducing the available active sites on the carbon surface for interactions with liquid and gaseous species during the SSR test and reducing the mass loss of the carbon anode. This hypothesis will be explored in future research. The effect of the origin of the core suggests that variability in raw materials or anode thermal history between the two industrial baked anodes may influence reactivity under SSR test conditions. The anode origin may capture some of this variability, but additional predictors must be added to improve the fit (for example, the coke's crystallite size or chemical composition). The root mean square error indicates the residual variability once the effect of density and the industrial anode's origin is considered. It can be viewed as a preliminary assessment of the method's repeatability. This metric will be refined in future work.

## 5. Challenges

During the study, some challenges were encountered that influenced the accuracy and reliability of the results. These challenges primarily involved the safety and security concerns and ensuring repeatability in experimental conditions and results.

The primary security concern is associated with the interaction between molten $Na_2SO_4$ and any moisture present, which can vaporize instantly and cause a deflagration. All the tools and parts to be in contact with the molten sulfate should be preheated at ~100 °C before the test. Also, as $Na_2S$ is a possible product of the reactions, exposure to humidity might cause the emission of $H_2S$.

Another concern in the SSR test could be the production of $SO_2$ and CO gases according to the reactions (2 and 3). The test must be conducted inside an enclosed, ventilated cabinet.

The probable splashes of liquid $Na_2SO_4$ during the withdrawal of the porcelain crucible and the pouring of the liquid on the steel pan posed another challenge. A tong with a minimum handling length of 300 mm is used for safely withdrawing the porcelain crucible. Suitable personal protective equipment should also be worn.

## 6. Conclusions

The proposed SSR test method has the potential to be used as a sensitive test method for carbon anode characterization and help assess the correlation between anode properties and their mass loss during reactivity with sodium sulfate. Initial experiments with lab-scale samples revealed limited sensitivity in mass loss results. Reliable and measurable mass losses were achieved, by using samples extracted from industrial anodes and adjusting the test temperature to 990 °C, for a test time of 30 min, and improving insulation to limit temperature fluctuations. However, the variation in baked apparent density among samples extracted from industrial baked anodes challenged the repeatability, and the uncontrolled thermal equilibration period after sample immersion reduced test sensitivity by allowing reactions before the $T_{test}$ is reached.

With the test conditions established, further tests on samples extracted from cores A and B showed a variation of mass losses (10–46) %. To explain this, an analysis of variance (ANOVA) was performed using centered apparent density and anode origin as predictors. Both factors showed a significant correlation with the mass loss ($p = 0.0007$ and $p = 0.0387$), with a global $R^2$ of 0.870 and a root mean square error of 4.37 %. These results demonstrate the SSR test's sensitivity to

anode structural differences and suggest its use as a complementary method for anode reactivity evolution.

## 7. Future Work

For the next steps of this work, it is essential to evaluate the repeatability of the SSR test to establish the reliability and robustness of the method. For this purpose, cathode samples will be used instead of carbon anodes. The use of cathode materials is motivated by their more homogenous structure, which minimizes the influence of microstructural variability and allows a more accurate assessment of test repeatability.

Future experimental work will focus on elucidating the effect of the time to reach $T_{\text{test}}$ on anode mass loss. Additional investigations will explore the influence of other test parameters, including larger sample sizes and larger amounts of sodium sulfate. Furthermore, the effect of anode properties, including porosity, crystallite size ($L_c$), chemical composition, and BT, on the SSR test results will be studied, and results will be compared to those of the air and $CO_2$ reactivity standard tests.

A thermodynamic approach will also be used to investigate the equilibrium state of the system. Several chain reactions are proposed in the literature to describe the overall reaction of sodium sulfate with carbon. Identifying the most prominent reaction may help better understand the possible correlations between test results and anode characteristics and evaluate the effects of factors such as temperature variations, phase changes, and the formation of secondary compounds on the SSR test results. In addition, kinetic study of the reactions and off-gas analysis can help to link the rate of different reaction pathways to the mass loss of the carbon anode samples.

## 8. Acknowledgements

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