IN-LINE SALT FLUXING PROCESS:
THE SOLUTION TO CHLORINE GAS UTILIZATION IN CASTHOUSES

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Abstract

Over the past decades, the aluminium industry has been very active in developing and implementing metal treatment technologies such as the TAC and RFI to reduce the negative environmental, health and safety (EHS) impacts of gaseous chlorine. However, most casthouses are still relying on the utilization of chlorine gas for in-line metal treatment. This requires maintaining an active chlorine network at considerable costs and EHS considerations.

A new technology developed at the Alcan Arvida Research and Development Centre in collaboration with la Société des Technologies de l'Aluminium du Saguenay (STAS) was implemented at the Alcan Iceland Ltd (ISAL) casthouse. This new technology has proven successful in achieving a complete elimination of chlorine and coping with the ultimate Alcan chlorine-free casthouse objective and commitment towards sustainable development.

This paper reviews the challenges to substitute chlorine gas utilization while achieving equivalent or better metallurgical performances and product quality. Preliminary metallurgical performances (hydrogen, alkali and inclusion removal) from pilot scale and industrial conditions are discussed. The new in-line salt fluxing process is described in terms of equipment and process requirements. Finally, the work of the Alcan ISAL casthouse towards total elimination of chlorine usage is presented.

Introduction

The positive impacts brought by the use of chlorine gas in the treatment of molten aluminium alloys are well recognized and have been reported in several publications [1-9]. Over the years, chlorine injection, mixed with an inert gas, has been applied at all steps of molten metal processing from molten metal pretreatment, furnace fluxing or in-line treatment (see Figure 1). Chlorine chemically reacts with alkali elements (Ca, Na, Li) by removing them by the formation of their respective chloride salt ($CaCl_2$, NaCl, LiCl). The presence of chlorine eases the removal of the non-metallic inclusions present in the melt by modifying their wetting properties, thus allowing their separation at the melt surface. The contaminants removed during treatment are known to increase the occurrence of defects such as edge cracks, tearing or corrosion, during the subsequent transformation of aluminium cast products.

Despite its beneficial impact, chlorine gas represents a major threat to health and safety of casthouse workers [10], surrounding communities and to the environment. Safe handling, storage and use of chlorine require a wide range of equipment and procedures to minimize risks. Major costs are associated with state of the art equipment such as chlorine chambers, neutralization systems and detectors. Preventive maintenance, training and emergency plans also involve resources and costs.
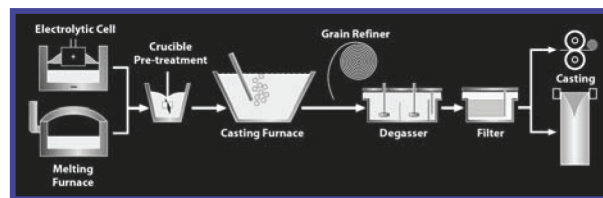

Figure 1. Molten aluminium path from potroom to cast products. Chlorine is/was used for pre-treatment, furnace treatment and for the degasser.

With this information in mind, the motivation to develop and implement alternatives to chlorine has always been very high throughout the aluminium industry [7, 9, 11-12]. While alternatives to chlorine are now successfully exploited for pre-treatment and furnace fluxing with technologies such as the TAC [13] and RFI [14], gaseous chlorine is still used for in-line metal treatment in most casthouses.

Operation of sealed in-line metal treatment units without chlorine is possible, but at lower or inconsistent alkali and inclusion removal efficiencies [15]. While feasible, this solution is unacceptable for the production of high quality value-added products. Over the years, several potential alternatives have been studied or tested [12-14, 16, 17], including: freons, halogenated hydrocarbons and fluoride-containing gases such as SiF_4 and SF_6. Injection of solid fluxes, although not extensively tested for in-line treatment, is very successful for furnace treatment as proven with the RFI technology. Its application for in-line metal treatment, while challenging, appears as the most promising option to replace chlorine [18].

This paper presents the on-going collaborative efforts of Alcan and STAS to develop and implement a chlorine-free in-line metal treatment process that will make possible the elimination of gaseous chlorine utilization in casthouses without compromising metal quality.

Principles and Process Description

To determine the potential of the identified alternative, the injection of MgCl₂ / KCl salt blends was tested on the Alcan Compact Degasser (ACD). The use of magnesium chloride based salt is not new to the aluminium industry. MgCl₂ / KCl fluxes are successfully injected using the RFI to treat molten metal in the casting furnaces. This salt has proven its efficiency in removing inclusions and alkali from the melt. However, application of salt injection to in-line treatment has to take into consideration the limited residence time of metal, the metal temperature, the gas flow rate to maximize degassing, salt dispersion and the kinetics of reaction of liquid salt.

Due to its conception as a trough-based in-line treatment process, the ACD has a typical residence time between 25 and 45 seconds. To reach equivalent metallurgical performances, when compared to chlorine, the salt flux has to be efficiently dispersed to maximize the interfacial area for the reactions with the impurities to be removed. The ACD was developed so as to maximize the total contact area of argon and chlorine bubbles with the melt [19]. This was made possible by the use of multiple high-speed radial injectors creating high shearing zones and very effective micro-bubble dispersion.
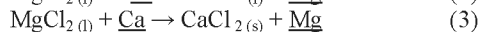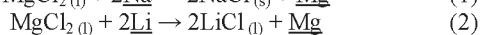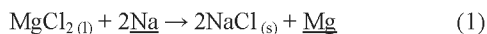
The kinetic factors which control the process have to be considered. It is well known that mass transport in the liquid phase is the rate controlling step for the alkali removal process [20]. The mass transport of alkali elements being the same for both chlorine gas and salt flux ACD, any variation in the kinetics of reaction would rely on the specific reaction mechanism.

In the case of chlorine fluxing, the concentration of chlorine is the same for each gas bubble, and the surface area available for the reactions will be proportional to the total surface area of the micro-gas bubbles generated by the rotors. However, for the salt injection process, the dispersal of the two-phase gas-liquid salt system will be affected by the interfacial tension of the gas-salt-metal system [21]. The total surface area available for the reaction will be different from the gas-gas system. Moreover, the kinetics of reaction will be affected by the reaction products formed at the surface on each salt droplet and the diffusion in the liquid salt phase [20].

Therefore, to ensure a successful conversion from in-line chlorine gas injection to in-line salt flux injection, several key factors have to be taken into account and the best combination of operating parameters identified.

Key Parameters Evaluated

Chemical Composition of the Reactant. Magnesium chloride is an active agent to remove impurities during metal treatment. Similarly to chlorine gas, MgCl₂ acts as a de-wetting agent to remove inclusions and chemically reacts with alkali, as shown by equations 1 to 3.

$$MgCl_{2\,(l)} + 2\underline{Na} \rightarrow 2NaCl_{(s)} + \underline{Mg} \tag{1}$$
$$MgCl_{2\,(l)} + 2\underline{Li} \rightarrow 2LiCl_{(l)} + \underline{Mg} \tag{2}$$
$$MgCl_{2\,(l)} + \underline{Ca} \rightarrow CaCl_{2\,(s)} + \underline{Mg} \tag{3}$$

To reduce its melting point (714 °C), pure magnesium chloride is mixed in different proportions with potassium chloride (KCl). Table I shows different salt compositions with their associated melting temperature. Compromises have to be established to find the adequate salt composition for in-line injection. Mixtures with high melting points may be difficult to melt during injection and may result in issues such as shaft blockage or solid particle carry-over. On the other hand, salt injection being performed in-line, it is preferable to inject the lowest amount of inactive material (KCl) to increase the kinetics of reaction as well as to reduce dross production or undesirable by-products in the melt after the degasser.

Table I. Melting Point of Different MgCl₂/KCl Blends

| Salt Composition	Approx. Melting Point
40% MgCl₂ – 60% KCl	430 °C
60% MgCl₂ – 40% KCl	485 °C
75% MgCl₂ – 25% KCl	570 °C
90% MgCl₂ – 10% KCl	660 °C
100% MgCl₂	713 °C

Salt Granulometry. Alike salt chemical composition, salt granulometry has an impact on the kinetics of reaction, which is primordial for in-line applications considering the relatively low residence time compared to furnace treatment. For this reason, the particle size of the salt used for furnace fluxing, such as the RFI, would not be appropriate for in-line applications.

Residual Moisture. Magnesium chloride is a hygroscopic compound that absorbs moisture if exposed to ambient air. Hydration occurs under different forms (MgCl₂·nH₂O, n=1, 2, 4, 6, 8, 12) depending on the temperature [22].

Moisture absorption by MgCl₂ has several detrimental effects. First, it increases the risk of explosion upon injection in molten aluminium. Second, hydrolysis reaction may occur, as shown in equation (4), and decreases the concentration of active compounds reacting with alkali elements [12].

$$MgCl_2 \cdot H_2O \rightarrow MgOHCl + HCl \tag{4}$$

Finally, residual moisture decomposition may increase the level of dissolved hydrogen in the melt, according to the following reaction:

$$2\,Al_{(l)} + 3\,H_2O_{(g)} \rightarrow Al_2O_{3\,(s)} + 6\underline{H} \tag{5}$$
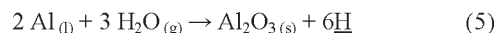
Since the main function of degassers is to remove hydrogen in the melt, the presence of residual moisture in the salt is certainly not suitable. The residual moisture content of commercially available salt mixtures was evaluated using the Karl Fisher method. Preparation under controlled atmosphere of salt mixtures of different proportions of MgCl₂ was also performed to minimize the risks of contamination by ambient moisture. These salts were then tested at pilot scale and during plant trials.

Experimental

Experimental Conditions

Based on the previous information, various blends of magnesium chloride and potassium chloride were chosen and tested during in-line salt fluxing trials performed at pilot and industrial scale. Tests

were performed on ACD units having 2, 4 and 6 rotors. These units have been modified to allow injection of a salt flux (the principle of salt injection being illustrated in Figure 2). Salt was injected using a salt injector prototype unit developed by STAS and maintained under inert atmosphere.
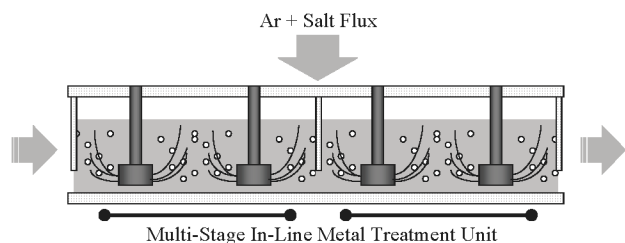

Figure 2. Principle of salt injection in the Alcan Compact Degasser (ACD) unit. Argon was used as the carrier gas.

Salt composition, salt injection rate, alloy composition and metal flow rate were varied during the trials. Argon was used as the carrier gas. Graphite rotors, having a larger internal diameter, were used to minimize risks of shaft blockage. Over all the tests performed, rotor blockage occurred only once due to a combination of a high salt flow rate and a high melting temperature salt mixture.

Trials were performed on pure, foundry, extrusion and sheet alloys at metal flow rates varying from 250 to 1000 kg/min. AlSCAN, PoDFA and horizontal disks, for OES measurements, were taken before and after the ACD to evaluate hydrogen, inclusion and alkali removal efficiency respectively.

Metallurgical Performances

Hydrogen Removal

As mentioned previously, hydrogen removal is one of the most important function of an in-line degasser as this is the only place in the processing chain to remove dissolved hydrogen. Therefore, any alternative that would impact the degassing performances will affect the hydrogen content of the final product. Due to its hygroscopic properties, $MgCl_2$ based salt could potentially increase the hydrogen content of molten metal.
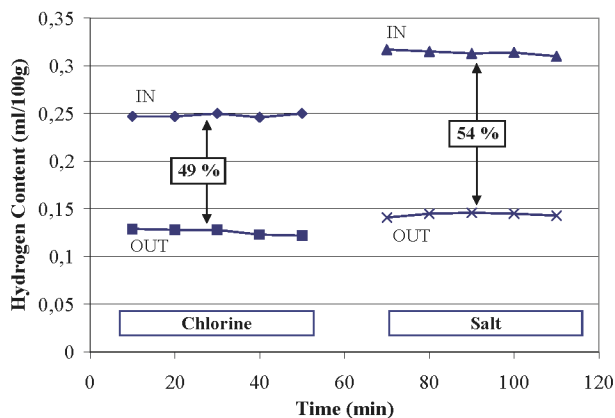

Figure 3. Hydrogen removal efficiencies in the ACD using chlorine and salt fluxing (argon flow rate 0.70 l/kg).

During the trials, the hydrogen level at the inlet of the ACD varied from 0.3 to 0.5 ml/100 g, and the outlet concentration was maintained below 0.2 ml/100 g. As illustrated in Figure 3, equivalent removal performances were achieved for salt injection compared to chlorine. The efficiency reached varied between 45 and 65%, which is similar to what is normally achieved using chlorine injection. The salt flow rate did not affect the hydrogen removal efficiency and the final content after the ACD.

Alkali Removal

Although most alkali removal is performed upstream during pretreatment and/or during furnace fluxing, several casting centers are relying on in-line fluxing to meet the final product specifications. During the trials, the incoming calcium and sodium contents varied from 0.5 to 5 ppm and from 1 to 15 ppm respectively. Figure 4 shows the relative sodium removal efficiency for chlorine and salt injection. As a comparison, the performances obtained without injection of a chemically active reactant (argon only) are also included in the figure. A 20% efficiency was reached using argon only due to the turbulent mixing conditions and vaporization of sodium at the metal surface. Indeed, the high activity coefficient (γ°_{Na} = 293) and the vapour pressure (P_{Na}=1 atm) [23] enable sodium removal in the ACD. However, this mechanism is less likely to occur for calcium (γ°_{Ca}<1, P_{Ca}=10^{-8} atm) [12, 23].

A 60% average removal efficiency was reached for chlorine injection. An average efficiency of 55% was achieved for salt injection added at a higher stoechiometric ratio, compared to chlorine, to compensate for a slower kinetic rate. The higher salt injection rate therefore allowed reaching alkali removal performances equivalent to chlorine. Overall, the alkali removal efficiency (for calcium and sodium) achieved varied from 40 to 90%.
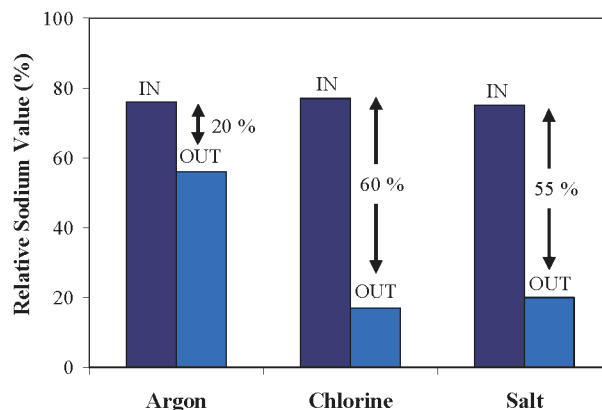

Figure 4. Alkali removal efficiency for argon only, chlorine and salt injection.

Inclusion Removal

An excellent inclusion removal efficiency was achieved for all tests performed using in-line salt injection (see Figure 5). The typical inclusion content at the degasser inlet varied between 0.1 and 0.8 mm^2/kg, as measured by PoDFA, and the inclusions found were mostly aluminium carbides (Al_4C_3), oxides (Al_2O_3, MgO) and spinel crystals.

This performance represents an improvement compared to the typical removal efficiencies generally obtained with in-line chlorine injection which may vary from 25 up to 60%. These superior performances may be associated to the difference in morphology of the chloride salt particles mechanically dispersed in the melt during salt injection compared to the chlorides formed in-situ during gaseous chlorine injection. It is believed that larger salt particles will have a greater ability to interact with the non metallic particles and modify their interfacial energy. In addition, the salt injection procedure and the flotation taking place inside the in-line treatment unit may enhance the separation process.
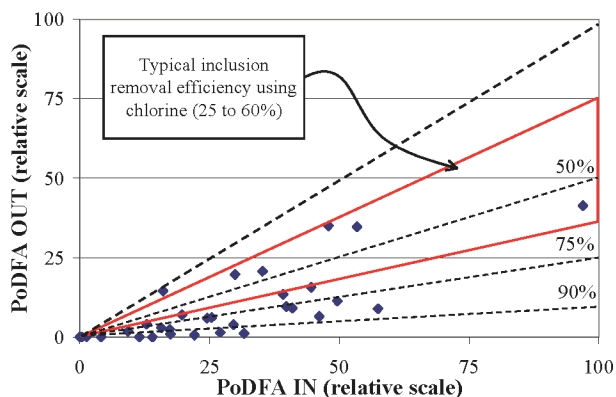

Figure 5. Inclusion removal efficiency for in-line salt injection process.

Although chlorides have a beneficial effect on metal cleanliness, chloride carry-over after the ACD is undesirable as it can be a major concern for potential defects in the final product [16]. An analysis of potential chloride salt carry-over in the metal flow outside the ACD was investigated.

Chloride salt concentration was measured on metal samples taken after the ACD using radiochemical neutron activation analysis and by metallographic analysis followed by EDX analysis under SEM, after dry polishing of samples. The radiochemical neutron activation analysis showed that the chloride concentration was proportional to the amount of salt injected. In general, the chloride concentration, immediately after the ACD, remained below 1 ppm, which corresponds to the average level typically measured with chlorine fluxing. The concentration of chlorides decreased with the sampling distance after the ACD, which indicates that chloride salt separates from the molten metal.

Table II presents the metallographic analysis results of argon, chlorine and salt fluxing samples. According to these results, the number of chlorides measured from the in-line salt fluxing process was lower than that of chlorine fluxing. As mentioned earlier, the morphology and the size of the chloride salt particles are probably key factors.

Table II. Number and Maximum Size of Chloride Inclusions Found in Sheet Alloys Treated With Argon Only, Chlorine Gas and In-Line Salt Injection

Injected	Avg. No. of Chlorides ($/cm^2$)	Max. Chlorides Size (μm)
Argon	0	0
Chlorine	1.0 ± 0.3	20
Salt	0.5 ± 0.4	20

Dross Condition

The condition of the dross formed inside the ACD unit, during the trials, was qualitatively evaluated. The dross produced using salt injection was dry and non-reactive. The amount of dross produced using salt injection was equivalent to that of chlorine injection.

In-Line Salt Injection Technology Development

Based on the very good metallurgical performance and numerous plant tests discussed in the previous sections, STAS has developed an initial industrial unit design to be used on a daily basis in casthouses. A first generation of the industrial version of the STAS In-line Flux Feeding System was elaborated to be installed on an ACD unit.

Description of the Equipment

The main function of the flux feeding system is to deliver a fluxing agent into molten aluminum at a given steady rate. The solid flux flow is regulated and controlled by a flux feeding unit that is mounted in a sealed housing which permits the salt to remain dry due to the supply of argon. This setup helps eliminating the problems usually associated with moisture absorption by the solid magnesium chloride-based fluxes.

The process gas is injected into the housing through a gas panel that controls the gas flow dedicated to the delivery of the flux. The gas exits through the same outlet as the solid flux to provide the appropriate gas-flux mixture. The latter is then supplied directly to the ACD hollow rotor shafts, which deliver the gas-flux mixture underneath the metal surface and within the high shear area generated by the spinning nozzle. Due to its melting point, the salt liquefies when submerged into molten aluminium, and the spinning nozzles disperse the salt droplets throughout the liquid aluminium. The gas flow is controlled by a mass flow controller and is set at 35 to 45 SLPM, which is the typical gas flow range for an ACD rotor.
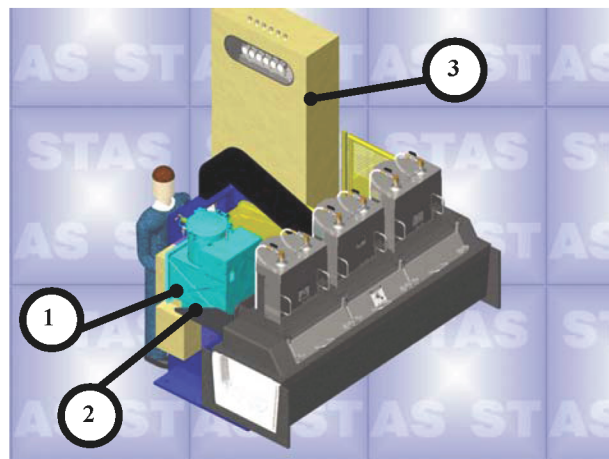

Figure 6. Schematic view of an ACD equipped with the in-line salt injection unit.

Figure 6 shows the mechanical components of the STAS In-line Flux Feeding System. A complete system is composed of:
- a flux feeder housing (item #1);
- the necessary structural support for attachment to the ACD (item #2);

- a gas panel and the isolating valves (item #3);
- gas-solid feeding pipes (not shown);
- an access and maintenance platform (not shown);
- an electrical power and control panel (not shown).

The salt injection unit is mounted on the back of the ACD unit and has no relative movement to the rotor modules. A structural support links both pieces of equipment. A rigid metallic tubing (not shown) connects the outlet of the flux feeder to the rotary joint located on top of the injection modules.

The flux feeder housing is fitted at about the same height as the ACD cover. Given its relative height, the flux feeder is easily accessed for refilling and maintenance by the use of a platform installed around the unit. In most cases, no major modifications are required on any existing ACD to convert from chlorine usage to solid salt injection. For retrofit on existing units, additional electrical and pneumatic panels are provided. On the other hand, for new units designed with salt injection capabilities, the ACD electrical and pneumatic panels will include the necessary hardware to control and to operate the STAS In-line Flux Feeding System. In all cases, integrating solid salt injection to Alcan Compact Degassers will be fairly straightforward.

In-Line Salt Technology Implementation
Alcan Iceland: A Chlorine-Free Casthouse

Located on the southwest peninsula of Iceland, close to the capital city of Reykjavik, the Alcan Iceland Ltd (ISAL) plant was built 40 years ago by Alusuisse. With an annual capacity of 180 kt, the ISAL casthouse produces a large array of sheet ingot alloys in the 1xxx, 5xxx, 6xxx and 8xxx series. A large part of ISAL's production is intended for critical applications such as foil, lithographic sheet, form pack (pharmaceutical, cosmetics) and automotive body parts. ISAL's focus is therefore to produce high quality metal to meet and even surpass customers' requirements.

Since its foundation, ISAL has undergone several expansions to keep up with the development of the potline: enlarging of furnaces, automating and maximizing the utilization of the existing equipment and others.

In 2003, as part of a complete reorganization of the casthouse to maximize productivity and reduce costs, every step of the process was analyzed. During this exercise, one very important question was raised regarding the use of chlorine in the process. Conscious of the risk associated with the use of chlorine for the workers, and given the complexity to import chlorine to the plant, ISAL decided to remove chlorine from its process. Although this decision was positively welcomed by the workers and ISAL's customers, the necessity to maintain the metallurgical performances was, for the casthouse, the main challenge. Indeed, as Figure 7 illustrates, chlorine was used in all the steps of metal treatment, from pre-treatment of liquid metal in crucible to in-line degassing. Since 2003, the total chlorine consumption has varied from 19 to 22 tons annually.

In 2005, ISAL's objective for a chlorine-free plant had started to materialize with the elimination of chlorine utilization for crucible treatment. Tests were also performed to evaluate the conversion of the crucible station to a TAC station, and to transform the rotary Gas Injection (RGI) unit to RFI, both technologies being well implemented in the aluminium industry. The RFI was

implemented for all furnace treatment, early in 2006, which reduced the chlorine consumption by about 17 tons annually.
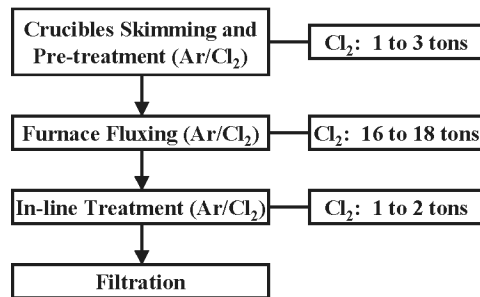
Figure 7. Metal treatment mapping in ISAL and associated annual chlorine consumption (2003 to 2005).

Nevertheless, a complete elimination of chlorine utilization at the ISAL casthouse was not possible until a solution for in-line metal treatment was available from the ARDC. The first trials with salt injection in an ACD were planned and performed very rapidly since the conversion from chlorine gas to solid flux was simple. Critical products in the 1xxx series were cast during the trials and the ingots produced were shipped to the customers for evaluation. The feedback received was positive. A second campaign was then planned for further evaluation. Trials were performed with salt injection in an ACD and in an RFI on multiple products having high metal quality requirements in the 1xxx, 5xxx and 8xxx series alloys. Once again, the feedback obtained from the customers was very good and confirmed the great potential of this technology to replace chlorine gas utilization for in-line metal treatment. Following the excellent metallurgical performances and the positive customer feedback obtained, the STAS In-line Flux Feeding System was permanently implemented on the ACD. As seen on Figure 8, the chlorine consumption of ISAL's casthouse went from 20 tons in 2005 to a total elimination within only two years. ISAL's objective to become a chlorine-free plant before the end of 2006 was then reached.
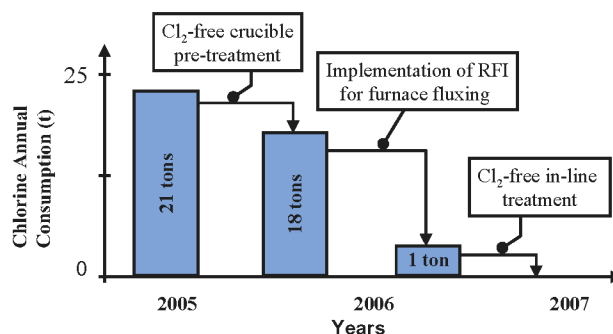
Figure 8. Evolution of chlorine consumption in ISAL. The chlorine-free casthouse objective was reached in 2006.

Conclusion

A new in-line metal treatment process based on salt injection was successfully developed at the Alcan Research and Development Center as a final step towards a complete elimination of chlorine utilization for metal treatment in casthouses.

Metallurgical performances achieved showed equivalent or better metal quality when compared to gaseous chlorine treatment.

An industrial version of the equipment fully retrofitable on the current ACD technology was designed by STAS and successfully installed at the Alcan Iceland Ltd. (ISAL) casthouse.

This development is a major step forward that confirms Alcan's leadership towards a sustainable development of the aluminium industry.

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