INCLUSION ENGINEERING - A KEY TOOL IN ACHIEVING STEELMAKING OPERATIONS STABILITY AT BHP WHYALLA

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Abstract
A new billet casting machine and ladle metallurgy furnace were commissioned at BHP’s Whyalla steelworks in June and October of 1999. The production of low Mn/Si ratio grades using, at first, an oxygen reheating station, and then later a ladle metallurgy furnace, provided many challenges to the achievement of stable steelmaking and casting operations. The Multi Phase Equilibrium model as developed by CSIRO has been used to solve complex metallurgical problems of practical importance in the production of silicon killed steels. Subtle changes to ladle practice for dissolved oxygen content combined with calcium treatment dictates inclusion composition and liquidus. Inclusion composition control has been a key requirement for making both open cast product with good surface quality, and also the control of refractory stopper erosion for submerged cast grades.
**Introduction**

In June of 1999 the Long Products Division of the Broken Hill Proprietary Company Limited (BHP) commissioned a new billet casting machine at its Whyalla steelmaking site. The caster commissioning occurred some months prior to the closure of steelmaking operations at BHP’s Newcastle plant. A new ladle metallurgy furnace (LMF) was also built at Whyalla to supply steel for the billet machine, however, some key pieces of equipment from the Newcastle plant were to be reused in Whyalla. Consequently, the billet caster commissioning had to commence without the aid of a LMF. Instead, the existing oxygen reheat station (IRUT - Injection Refining Up Temperature) was used to supply steel for billets until the ladle metallurgy furnace became fully operational. As such, a somewhat unique opportunity to directly understand the impact of ladle treatment methods on inclusion control and process control was realised.

Successful billet casting requires a good control of ladle steelmaking practices so that specific deoxidation inclusion compositions can be achieved. In particular, liquid phase inclusions are generally considered a prerequisite for sustained metal flow through small section refractory nozzles (18 mm diameter). The IRUT ladle station at Whyalla was originally installed to supply aluminium killed steel to a combination slab/bloom caster, and, the inclusion chemistry is fairly simple. Supplying calcium treated silicon killed and aluminium killed steels to the billet machine was more challenging. This demanded a deeper understanding of inclusion control and modification to achieve smooth casting operations with metering nozzles, and later, stoppered submerged entry nozzles.

In developing IRUT and LMF practices for an extensive grade range at Whyalla, a combination of knowledge sources was utilised. CONCAST Standard AG supplied both casting machine and ladle furnace, including technical assistance during commissioning. The development and optimisation of ladle practices for both IRUT and ladle metallurgy stations was undertaken jointly by CONCAST Standard AG and BHP’s technical and operations people. Establishing the links between theoretical inclusion engineering, actual inclusion compositions, and acquired operational knowledge, was achieved by standard steel sampling and Scanning Electron Microanalysis techniques. The results were used both directly, and in combination with a Multi Phase Equilibrium model (MPE), developed by the Commonwealth Scientific Industrial Research Organisation (CSIRO) in collaboration with BHP and other Australian companies.

This work focuses on the production of calcium treated manganese/silicon killed steels at Whyalla. Inclusion composition control from tapping through to mould is discussed for IRUT and LMF practices, with a special emphasis on the link between inclusion composition and tundish stopper tip performance.

**Inclusions After Tapping**

Empirical recipes for alloy additions at tap have been evolved and enshrined in metallurgical folk law for most of the twentieth century. Occasionally, a direct link between operational practice and fundamental measurements can be made. In the billet casting of manganese/silicon killed steels it is generally known that trace amounts of aluminium in steel can have a very detrimental effect on castability, but this is rarely quantified in any detail. The importance of aluminium control and its direct relationship to inclusion composition was
elucidated in Whyalla by using the fundamentals based MPE model, and the direct sampling and analysis of inclusions in steel.

In Whyalla, inclusion analyses were generally obtained from routine lollipop production samples. At the base of the lollipop stem, the steel was generally found to be free of sampler deoxidant inclusions (zirconia) and suitable for investigation by microprobe analysis. Bomb samples were initially taken to study the inclusions, but this technique was soon discarded after finding that most of the necessary information could be obtained directly and conveniently from the lollipop samples.

In the early stages of caster commissioning, trial work was undertaken to determine the impact of aluminium additions at tap on the process, and on inclusion composition in particular. Figure 1 summarises results of the measured average inclusion composition after tapping and preliminary ladle stirring for several steel grades made at Whyalla. In general, the average compositions were determined from the larger sized inclusions (greater than 10 µm), which were taken to be most representative of those inclusions actually present under ladle treatment conditions. The smaller inclusions, which were observed in some bomb samples, generally contained a lower alumina content as a result of steel deoxidation reactions occurring in the sampler itself during cooling. An example of this is shown for heat 510065 in Figure 1, where the average Al₂O₃ content decreases from 41% for the larger inclusions (> 10 µm) down to 20% for the smaller inclusions (less than 5 µm). Even in the larger inclusions, it should perhaps be expected that some change to inclusion composition during cooling has occurred. So, an assumption of measured inclusion/metal in ladle equilibrium may not be strictly correct, and some deviation from laboratory measured slag/metal equilibrium is to be expected in some cases. However, the observations at Whyalla for tapping inclusions, suggest that a near equilibrium condition between metal and slag (averaged inclusion composition) is generally achieved. An example MPE calculation for the heat 512056 is shown in Figure 1. As the dissolved Al content in steel is increased along the line wxyz, the calculated steel oxygen content and equilibrium inclusion composition is indicated. At point z, the calculated alumina saturated inclusion composition is close to the measured result (hexagon point in Figure 1). The calculated dissolved oxygen content was 64 ppm versus an observed value of 52 ppm, which is considered a reasonable agreement with a plant measurement.

The Al₂O₃ content has a direct impact on the inclusion liquidus, which in billet casting generally determines whether a steel will cast or not. Inclusions that contain a significant amount of solid phases at tundish temperature are prone to produce nozzle blockage during casting. Figure 1 shows that when no aluminium is added at tap (heats 510956 and 510957) less than 10% Al₂O₃ in the inclusions is observed, most of which comes from the ferrosilicon added at tap. Increasing the aluminium addition at tap will increase the Al₂O₃ content of inclusions accordingly, subject to the dissolved oxygen in steel prior to tapping and the amount of reoxidation that occurs in the ladle during tapping. The oxygen balance in the ladle during tap is very sensitive to process variation. For example, heat 511462 in Figure 1, the inclusion population was entirely solid Al₂O₃ and clearly not desirable from the viewpoint of castability (dissolved oxygen 29 ppm). By comparison, heat 511418 which is the same steel grade, contained globular alumina saturated manganese silicates (dissolved oxygen 43 ppm).

The Al₂O₃ content of inclusions in Si/Mn killed steels that are present immediately after tapping can be inferred from the total Al analysis (Optical Emission Spectrometry). This is
because the dissolved aluminium content in metal is small in these steels with free oxygen greater than 30 ppm or so. Figure 2 shows the relationship between the total Al content of steel and the average Al$_2$O$_3$ content of inclusions observed after tapping.

Up to the point of Al$_2$O$_3$ saturation, the relationship between % Al$_2$O$_3$ and total Al content is linear. Beyond Al$_2$O$_3$ saturation, the same linear relationship is not followed. Tomioka et al$^2$ used a secondary ion mass spectrometry technique to measure trace levels of aluminium in silicon/manganese killed steels. They found that for liquid inclusions, the dissolved aluminium content was typically 1 to 5 ppm. Maeda et al$^3$ also reported results that showed the relationship between soluble Al content in silicon killed steel and the Al$_2$O$_3$ content of the inclusions. Their results are shown in Figure 2. About 10% of the total Al shown in Figure 2 can be attributed to dissolved aluminum in steel. Figure 2 can be useful in practice to indicate the likely inclusion composition immediately after tapping. For Si/Mn killed steels made through the IRUT ladle station at Whyalla, this information can be useful in assessing the prospects for castability in cases where oxygen reheating (which brings about a further change in Al$_2$O$_3$ content) is not necessary. Figure 2 also shows that the effect of sample preparation can be significant in the total Al determination. Silicon carbide and also alumina finishing papers were used to prepare some samples. Contamination from the alumina finishing paper was typically several ppm Al, which is significant, especially at less than 20 ppm total Al content.

**IRUT - Shrouded Casting Process Route**

In the initial commissioning phase of the billet caster, steel was made by the IRUT process for casting with either open or nitrogen shrouded tundish to mould streams. In order to minimise the impact on steelmaking converter refractory wear, a decision was made to oxygen reheat all billet steels at the IRUT. This was effective in maintaining a reasonable control on end of blow temperature, slag carryover, ladle station arrival temperature and ladle station treatment time. The typical compositional changes in averaged inclusion chemistry from tapping through to tundish are mapped in Figure 3. The grid size of the ternary diagram in Figure 3 is specific to the particular CaO content of inclusion. For example, where an inclusion contains 32% CaO, the full scale axis is 68wt% oxide and the corresponding grid size of the diagram is equal to 10 lots of 6.8wt% each. Any FeO in inclusions was included with MnO for the purposes of plotting inclusion chemistries on this diagram.

The inclusion trajectory begins at tapping where alloys, including some aluminium, were added to give in this typical case 52 ppm dissolved oxygen at 1621°C. The inclusions after tapping contain 42% Al$_2$O$_3$ and are close to saturation. After reheating the steel in the ladle by blowing oxygen and adding additional aluminium, the Al$_2$O$_3$ content of inclusions decreases to about 30%. The dissolved oxygen content and temperature in this case were measured at 95 ppm and 1629°C. The ladle was then calcium treated with CaSi wire to ensure trouble free casting. The CaO content of the inclusions is 30%, and the MnO content has decreased from 29.9% down to 1.7%. Finally, in the tundish, average inclusion composition is again measured and the CaO content is 22.1% and MnO 6.1%.

The positions of the solid/liquid phase boundaries drawn in Figure 3 show that all the measured inclusion compositions are liquid phase. The MPE model has been used to calculate inclusion liquidus for the measured average inclusion compositions. This is shown in Figure 4 at each of the steel processing stages. On arrival at the ladle treatment station (after
a preliminary ladle stir) the inclusion liquidus, in this case, was essentially the same as steel temperature. After reheating with oxygen the inclusion liquidus is 92°C less than steel temperature. The change in inclusion liquidus is mainly due to a decrease in the alumina content. After calcium treatment, the inclusion liquidus is 150°C less than steel temperature. The change in liquidus is due to the substitution of MnO in inclusions by CaO and the direct increase in CaO content that occurs via the reaction of dissolved oxygen and calcium. In the tundish, the inclusion liquidus is 84°C less than steel temperature. This is mainly due to the drop in steel temperature, but some change in the inclusion liquidus does occur as a result of fading CaO and increasing MnO content, which is a consequence of the combined effect of deoxidation, reoxidation and inclusion float out.

**Oxygen Control and Operations Stability**

In the initial stages of practice development for silicon killed low Mn/Si ratio grades, the casting was done with a nitrogen shrouded steel stream between tundish and mould. This system of reoxidation control is not always perfect, and an early lesson was learned, the hard way in Whyalla, for heat 511490. In this heat, a combination of insufficient calcium treatment and reoxidation in ladle and casting processes resulted in multiple strand breakouts. The accumulation of a viscous inclusion/reoxidation slag in the mould was observed. During the casting process, this type of high melting point slag can become trapped between mould wall and newly solidified steel layer and can culminate in a liquid steel breakout. Samples of the mould slag were recovered from both mould and billet surface and its compositional range is indicated in Figure 5. The CaO content was 2 to 12% and the SiO$_2$ content greater than 57%.

The composition of the mould slag lies at the silica saturation phase boundary where viscosity and liquidus temperature are both high.

Subsequent shroud cast heats were much more successful, where CaSi additions were increased in the IRUT ladle treatment to modify inclusions and control dissolved oxygen content. Heat numbers 512056 and 512289 are examples of tundish inclusion composition (Figures 5 and 4) where good billet surface quality and castability were achieved for the shrouded stream casting operation. The tundish inclusion liquidus in these heats was calculated at 1460 and 1480°C respectively, which is comfortably less than steel liquidus at between 1520 and 1530°C. Heat numbers 513124 and 513127 are examples where poor billet surface quality occurred (Figure 5). These samples were taken from entrapped slag in the billet surface and clogged nozzles respectively. In heat 513127, the nozzle deposit composition has a liquidus of 1700°C, which is well above tundish temperature. Insufficient Ca modification of alumina inclusions was achieved in this case, resulting in nozzle clogging at cast. In heat 513124, the mould slag composition recovered from the billet surface had a calculated liquidus which is very close to the steel liquidus. The combined effect of solid phase precipitation and increased viscosity of slag in the mould are likely causes for the entrapment of slag pockets during casting which culminates in a poor billet surface condition.

**LMF - Submerged Casting Process Route**

Where good surface quality and general steel cleanliness is required in rolled product, submerged casting between tundish and mould is generally practiced. At Whyalla, a stoppered tundish system is used to control metal flow for steel grades that require submerged cast operations. In submerged casting, inclusion and mould slag composition control is less of an issue for achieving good billet surface quality. However, the need to minimise erosion of
refractory stopper tips is very important to maintain metal flow control. Oxygen control and hence inclusion composition, again has a major impact on operational stability. Always there is a balance between nozzle clogging and refractory erosion and sequence length. While it was possible to make the submerged cast low carbon, low Mn/Si ratio grade through the IRUT process, operational stability was not good. In particular, tundish stopper tip wear (magnesia-carbon refractory) was erratic, which made it difficult to reliably make more than 2 or 3 heats in a sequence as shown in Figure 6. The main problem at that time was thought to be reoxidation from the ladle slag after calcium treatment. This made it very difficult to consistently control the dissolved oxygen content in tundish steel at less than about 30 to 35 ppm.

Some months after caster startup, the ladle metallurgy furnace (LMF) and slag raking station were commissioned. In this process route, carryover converter slag was completely removed from the ladle before rebuilding a new low FeO+MnO top slag. An immediate improvement in ladle and tundish oxygen control was possible with the LMF. A dissolved oxygen content in tundish steel of typically 20 ppm made it possible to increase sequence length to a monthly average of 4.1 heats per tundish (Figure 6). Tundish stopper tip performance for the LMF process route, was however, still erratic, and a problem for operations stability and scheduling. Inclusion composition was again checked in order to get a deeper insight into the new process route.

Based on the knowledge from the IRUT process route, where some relationship between total Al in steel and Al$_2$O$_3$ content of inclusions was generally observed (Figure 2), two heats were selected for closer investigation of inclusion composition. They were heat numbers 514583 and 514693, which had total Al contents in tundish samples between 20 and 30 ppm, and less than 10 ppm respectively. The averaged inclusion compositions from mid-ladle tundish samples are shown in Table 1. The inclusion size was small and in the range 5 to 7 µm, so some high FeO results, which were a likely outcome of electron beam interference with the surrounding iron matrix, were not included in the averaged results shown in Table 1. The number of inclusions included in the averaged results are shown in the Table. A tundish inclusion analysis from the IRUT process chain (heat 512289) is given for comparison. In changing the ladle treatment operations, the inclusion composition has been altered considerably. For the LMF treatment, the CaO content in inclusions is much higher and the MnO content is now close to zero. In the LMF process route a cleaner steel is to be expected due to better ladle slag composition control and gentle bottom stirring of the ladle. So, clearly, for an equivalent input of CaSi wire, the extent of calcium modification to inclusions is much greater. The resulting lower MnO content in inclusions is a fortunate consequence in terms of refractory stopper tip wear.

**Inclusion/Stopper Refractory Reaction**

It is well known that slags with high FeO and MnO content will attack alumina and magnesia based refractories$^4$. In the context of tundish stopper tips, a reaction with inclusions should also be expected. While many mechanisms can be responsible for refractory erosion, it is likely that the oxygen contained in inclusions and dissolved in the steel constitutes the main mechanism of attack by reaction with carbon in the refractory matrix as described by reactions 1 and 2.
\[ [O]_{\text{steel}} + C_{\text{refractory}} = CO(g) \quad \text{reaction 1} \]
\[ (\text{MnO})_{\text{inclusion}} + C_{\text{refractory}} = [\text{Mn}] + CO(g) \quad \text{reaction 2} \]

In changing the ladle process route from IRUT to LMF at Whyalla, a decrease in tundish oxygen and a decrease in the inclusion MnO content were both observed. A general improvement in stopper tip wear rate was observed and could be understood fairly readily on the basis of inclusion composition.

**Tundish Inclusion Liquidus**

Using the MPE model, inclusion liquidus was calculated for heats 514583 and 514693 at 1440°C and 1510°C respectively. Such a large difference in liquidus was not really expected, since the inclusion CaO content was fairly similar in both heats. The Al\(_2\)O\(_3\) and SiO\(_2\) contents were however quite different. Because the Al\(_2\)O\(_3\) content of the inclusion can be controlled to some extent by the dissolved oxygen and aluminium in steel prior to calcium treatment, liquidus calculations were examined for inclusion compositions with higher and lower Al\(_2\)O\(_3\) contents around the measured points. This was also done for the tundish inclusion composition from the IRUT process route and is shown in Figure 7. For the IRUT case at constant 22.1% CaO, an increase in Al\(_2\)O\(_3\) content brings about an increase in inclusion liquidus as might be expected. For the LMF case at constant 41.5% CaO, a minimum in inclusion liquidus is calculated at 41% Al\(_2\)O\(_3\). So, in this high CaO content case, a simple increase in dissolved Al content (and decrease in dissolved oxygen content), may not bring about the desired effect of accreting inclusions at the stopper tip, but rather, the reverse is likely to occur in some instances.

For an operating plant, such a fine balance with Al\(_2\)O\(_3\) control in inclusions is not easily guaranteed, so a different operating window for inclusion composition was explored. Clearly, at 22.1% CaO in the inclusions the oxygen content of both steel and inclusions is too high to constrain the reactions with carbon in refractory. At 41.5% CaO in inclusions, the oxygen content of the steel + inclusion system is low enough, but the inclusion liquidus is too sensitive to process variability in Al\(_2\)O\(_3\) content. Logically, the CaO content of inclusions between 22.1 and 41.5% was investigated. It was possible to make an intuitive selection of a tundish sample that contained about 30% CaO in inclusions on the basis of the total Ca content analysis for steel. Figure 8 shows the relationship between total Ca content of steel and CaO content of inclusions for low Mn/Si ratio, low C steel sampled at the tundish. In silicon killed steels, where the dissolved Ca content of steel is low, the total Ca content of the steel should be proportional to the CaO content of inclusions as inferred from Figure 8. The variability in the measured total Ca content of steel is expected to be influenced by steel cleanliness. Two heats with the same CaO content in inclusions can have different total Ca content measurements due to a difference in the volume fraction of inclusions present.

Heat number 514940 contained 35 ppm total Ca in the tundish and was checked for inclusion composition. The averaged (and normalised) CaO content was 30.1%, and the MnO content 0.5% as shown in Table 1. The MPE model was again used to check the inclusion liquidus as a function of Al\(_2\)O\(_3\) content around the measured point. In this case at constant CaO content of 30.1%, a monotonic relationship between inclusion liquidus and Al\(_2\)O\(_3\) content is calculated as shown in Figure 9. At 30.1% CaO in tundish inclusions, the MnO content is still
low enough to minimise the reaction with carbon in refractory stopper tips, and, the Al$_2$O$_3$ content can be adjusted appropriately at the ladle according to erosive or clogging tendencies during cast. The liquidus temperature for tundish inclusions in heat 514940 was 1500°C.

As a result of changing both the Al$_2$O$_3$ and CaO contents of inclusions, the operational stability of the plant for making low carbon, low Mn/Si ratio grades is now much better. As of March 2000, the average monthly sequence length was 5.33 (Figure 6).

**Inclusion Trajectory for LMF Process Route**

The inclusion trajectory for the LMF heat 514940 is shown in Figure 10 in comparison to the IRUT trajectory. The ladle inclusions after tapping are similar in composition and lie near to the alumina saturation boundary. The dissolved oxygen content and temperature for the start of treatment IRUT and LMF process routes were respectively, 52 ppm at 1621°C and 39 ppm at 1588°C. The difference in dissolved oxygen content at treatment start is mainly due to temperature and its effect on oxygen solubility. The inclusion morphology at treatment start is globular, and the observed size range was 5 to 20 μm. Following temperature adjustment and alloy trimming, the inclusion trajectories for IRUT and LMF processes differ considerably. For the IRUT process, the inclusion Al$_2$O$_3$ content decreases as a result of oxygen reheating, whereas Al$_2$O$_3$ content increases for the LMF. In the LMF process, the inclusions prior to CaSi treatment were entirely alumina at a dissolved oxygen content in steel of 32 ppm. These inclusions are not globular, but rather, rounded in shape and 5 to 10 μm in size. Some clusters were also observed in the sample, their overall size being estimated at 40 to 70 μm.

After CaSi treatment (0.1 kg Ca/t steel) the CaO content of inclusions is 30.1% and the Al$_2$O$_3$ content 42.7%. The Al$_2$O$_3$ and CaO contents of inclusions are significantly higher than observed for the IRUT process route. Consequently, the dissolved oxygen content straight after CaSi treatment is lower at 10 ppm for the LMF process as compared to 36 ppm for the IRUT. In the IRUT process the inclusions after Ca treatment were globular and typically less than 7 μm in size. In a scanned area of sample of 38mm$^2$ there were 180 inclusions. In comparison, the LMF sample contained 10 inclusions, all globular and 5 to 7 μm in size.

In the tundish, the inclusion composition can change according to the amount of process reoxidation and the temperature dependence of deoxidation reactions. In the IRUT process route, the Al$_2$O$_3$ content of inclusions decreases as a result of dilution from new deoxidation products which are richer in MnO and SiO$_2$. In contrast, the Al$_2$O$_3$ content of the LMF inclusions between ladle exit and tundish increases. This is also due to new deoxidation products. In the LMF case the dissolved Al content is just sufficient to maintain an Al$_2$O$_3$ rich new deoxidation product as opposed to Al$_2$O$_3$-MnO-SiO$_2$ in the IRUT process route. The increase in Al$_2$O$_3$ and decrease in CaO should increase inclusion liquidus as inferred from Figures 7 and 9. In this particular instance, some tundish reoxidation can actually help to minimise tundish stopper tip wear by increasing inclusion liquidus. Of course, too much reoxidation will ultimately consume dissolved Al until the inclusion MnO content then begins to increase, as already noted for the IRUT process.

**Conclusion**
Inclusion engineering has been a key requirement for the successful billet casting of silicon killed steels at BHP Whyalla. CSIRO’s Multi Phase Equilibrium Model has proved to be a versatile tool for understanding complex steel deoxidation reactions with Mn, Si, Al and Ca. In this work, use of the MPE model to make simple liquidus calculations for measured inclusion compositions in shrouded and submerged pour casting has given a good basis to make process development decisions for steelmaking operations. A proper control of inclusion composition, to ensure castability and mould slag control, is necessary for achieving good surface quality in shrouded pour casting. In submerged pour casting, an improvement in tundish stopper tip wear has been achieved. Optimisation of inclusion composition by deoxidation and calcium treatment practices has been a key part of this success. It has often been said that if we take care of the slag, the steel will take care of itself. While we cannot disagree with this, we can add, that inclusions contain a wealth of steelmaking information that can be used to optimise operations. Taking care of the inclusions, has helped to solve complex metallurgical problems of practical importance in the production of silicon killed steels at Whyalla.

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References
Figure 1. Measured average inclusion compositions after tapping for Si/Mn killed billet steels at Whyalla.
Figure 2. The relationship between total Al content of steel and the average $\text{Al}_2\text{O}_3$ content of inclusions (>10 µm) after tapping for Si/Mn killed billet steels at Whyalla.
Figure 3. The CaO- Al₂O₃-MnO-SiO₂ oxide system at 1530°C showing the inclusion trajectory for the IRUT shroud cast process route.
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Figure 10. The CaO-Al$_2$O$_3$-MnO-SiO$_2$ oxide system at 1530°C showing the inclusion trajectory for the IRUT shroud cast process route in comparison to the LMF submerged cast process route.
Table 1. Average inclusion compositions (wt%) for mid ladle tundish samples from LMF heats 514583, 514693, 514940 and IRUT heat 512289

<table>
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<tr>
<th>Heat No.</th>
<th>No.</th>
<th>Al₂O₃</th>
<th>SiO₂</th>
<th>CaO</th>
<th>MnO</th>
<th>FeO</th>
<th>T_{liquidus}</th>
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<td>1440</td>
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