APPENDIX I MEFOS DATA



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HYMELT CAMPAIGN 1,

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by

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1 Introduction

The first test campaign in the development of the EnviRes HyMelt process has been performed at MEFOS. The tests have been made in the 6 ton universal converter equipped with facilities for injection of hydro-carbides and oxygen.

The technical objective was to make preliminary evaluation the process and functional test of the equipment and measuring systems.

The tests were made for:

- Solid feed injection for Illinois #6 (coal) and Pet-coke (coke)
- Liquid feed injection for 325 Aromatic extract (oil)
- Oxygen blowing
- Gas measurement
- Dust sampling
- Process control
- Process strategies

The campaign was carried out for six days in the period from June 5 to 13, 2003.

This document is a completion of collected data and a preliminary evaluation of the operational results and performance of process equipment and measuring systems.

2 Equipment

2.1 MEFOS standard equipment

2.1.1 Electric arc furnace

The electric arc furnace is a fully ceramic lined AC furnace suitable for melting 4 to 10 ton of steel or hot metal. In the project the furnace was used for production of high carbon iron melt in charges of 5 - 6 ton.

Technical data:

- Heat size 4 10 ton
- Trafo 4,9 MVA
- Shell diameter 2800 mm
- Diameter lined2100 mm

2.1.2 Universal converter

The experiments were carried out in MEFOS universal converter. The converter has a nominal max capacity of 6 ton and can be altered into a variety of converter pro??cesses for metal refining. In the experiment the converter was used as a 5,5 ton, top blown vessel with bottom purging of inert gas.

Technical data:

Shell diameter 2000 mm Diameter lined1410 mm Height lined 2893 mm Lining

Far impregnated magnetite Radex ST

Two hoppers above the converter were erected for slag former and cooling scrap addition.

Totally four top lances was mounted:

- Oxygen lance for decarburisation
- Lance for solid injection alternatively lance for liquid injection
- Sub lance for metal sampling and temperature measurements
- Gas suction lance for sampling of process gas from converter gas atmosphere

In the converter bottom, on the half radius from the wall, a bottom tuyere was mounted for inert gas or hydrogen purging.

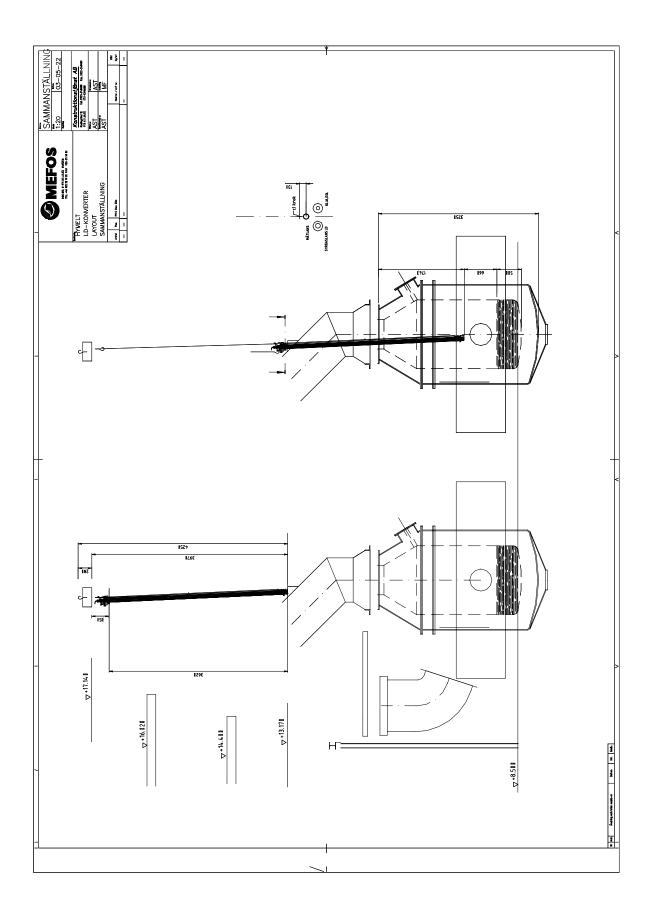


Figure - 1 MEFOS universal converter Hy-Melt, 5 ton, set up

The MEFOS venturie scrubber was used for gas treatment. Before gas cleaning the process gas has to be completely combusted by sufficient amount of air above the converter mouth.

Technical data, venturie scrubber: Gas flow rate 21 000 m³n/h Gas temperature inlet 650 $^{\circ}$ C Pressure drop across venturie 500 – 1900 mm wq

2.1.3 Material injection system

For injection of coal and pet-coke a 3 m³ powder dispenser system was selected. The system has advantages in flexibility regarding blowing conditions and allows handling and charging of fine grained material from "big bags" by use of suction.

Technical data: Dispenser volume 3 m³ Spec. transport gas ~0, 05 m³n/kg Outlet diameter variable Max pressure 10 bar

2.1.4 Measurements, sampling and automation

For process control a set of selected measurements were sampled and evaluated by an inhouse system for converter automation that runs on a LabView platform. The system includes, data logging, standard closed loop regulation and modules for real time evaluation of fundamental process parameters.

Measurements:

```
Gas supply: range
        Oxygen to lance, line C
                                          0 - 20 \text{ m}^3 \text{n/min}
                                                  0 - 1 \text{ m}^{3} \text{n/min}
        Nitrogen bottom purging, line D
        Nitrogen material transport 0 - 1 \text{ m}^3 \text{n/min} (Operator notes)
Combusted gas
                                 0 - 20000 \text{ m}^3 \text{n/h}
        Flow rate, venturi
        Composition:
                        0 - 5\%
                CO
                CO_2 = 0 - 20\%
                         0 - 23\%
                O_2
                SO_2
                        0 - 0, 5\%
Materials
        Dispenser weight
                                 0 - 3000kg
```

Bin weights	
Cooling scrap	0 – 1000 kg
Lump lime	0 – 1000 kg
Oil feed	0 – 20 kg/min

Oxygen lance pos mm above metal surface Injection lance pos mm above metal surface

Non-continuous measurements:

Metal temperature	$0 - 1800 \ ^{\circ}C$		
Metal composition			
Process gas composition con	ventional		
СО		0 - 100%	
CO_2		0 - 20%	
H_2		0 - 20%	
Process gas composition mass spectrometer MS 127			
H ₂	1	0 - 100%	
Process gas composition mas	ss spectrometer AIR SENSE 2000		
0 1	1		
H_2		0 - 100%	
H_2S		0 - 2%	
COS		0 - 100 ppm	
CH ₄		0 - 4%	

Special measurements and sampling not recorded on the process computer:

Metal weight, charging and tapping Analysis of sampled metal and slag Poured slag weights Dust sampling in converter for composition analysis Dust sampling in combusted gas for composition analysis and load Manual additions of Al-bars, FeSi, FeV and FeS Hydrogen gas for bottom purging

In blow metal sampling was made by use of a sub-lance system. Sampling during process stop in tilted converter was manually made. In both cases the samples were either analysed for carbon and sulphur by use of a Leco combustion analyser or sent to SSAB for spark emission spectrometry giving almost a complete analysis. All samples were stored for reference and further evaluations.

Slag sampling was manually made when the converter was tilted. All samples were stored and selected samples were analysed by X-ray at SSAB.

Dust sampling was made by two methods:

- 1. Process dust from the filter of the converter gas.
- 2. Iso-kinetic sampling of combusted dust from the duct.

Process parameters calculated on-line for supervising were:

- 1. Material injection flow rate
- 2. Gas flow rate, supply and combusted gas
- 3. Carbon balance for carburisation yield
- 4. Oxygen balance for oxygen yield during decarburisation
- 5. Carbon content in liquid metal

2.2 HyMelt equipment

2.2.1 Injection lance for coal and pet-coke

A water-cooled lance for solid material injection was designed and constructed. Technical data: Total length 4800 mm Diameter 76 mm Outlet nozzle diameter 7,0 mm, other dimensions were also tested

For transport of material from dispenser to the lance a flexible rubber hose was selected. The hose quality has reasonable abrasive wear resistance and can, with proper heat protections, be used in metallurgical environment.

2.2.2 Injection lance for oil feed

A water-cooled lance for oil injection was designed and constructed.

Technical data:Total length4800 mmDiameter76 mmOutlet nozzle diameter1,9 mm

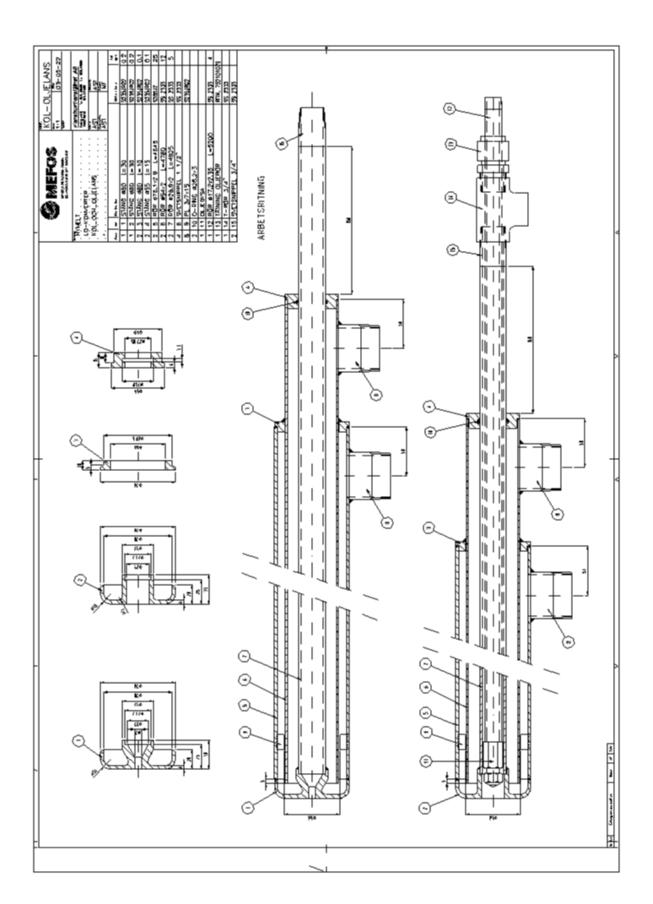


Figure 2 - Water-cooled injection lances for solids and oil

2.2.3 Injection system for aromatic extract

A special designed oil injection system was set up for low viscosity oil. The demand for short transport distance of the oil called for special safety arrangements and it was found that the storage tank with heating and the pump were to be mounted in a steel container. The set-up gives sufficient safety for spitting and slag splashing that accidentally generates from oxygen converters.

Technical data: Storage volume 3 m³ Max pressure 22 bar Nominal oil feed rate 10 kg/minute

2.2.4 Lance for oxygen blowing

The oxygen lance was designed as a water cooled single Laval nozzle for BOF converters.

Technical data: Total length 4800 mm Diameter 76 mm Nominal flow rate 10 m³n/min Outlet nozzle diameter 15, 5 mm Mach No 2

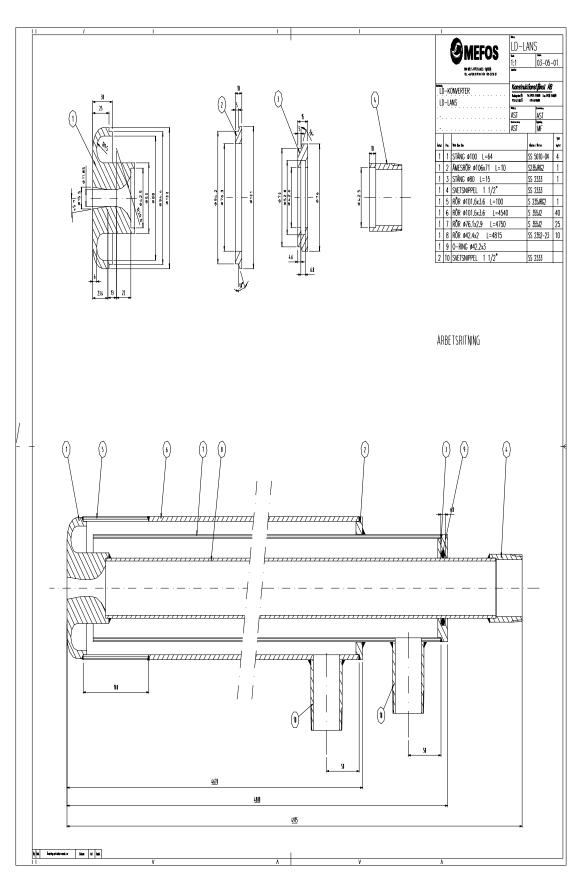


Figure 3 – Oxygen lance

2.2.5 Bottom purging tuyere

Top blown converters suffer from low metal stirring resulting in low mass transport rates witch thereby sets a limit for material trough put. A simple and efficient solution is to install a gas purging element in the bottom of the converter. For the experiment a double eccentric pipe tuyere was selected for injection of nitrogen and hydrogen.

Technical data: Length 700 mm Outlet slit, diameters 17,40 - 17,95 mm Nominal gas flow rate 0,5 m³n/minute

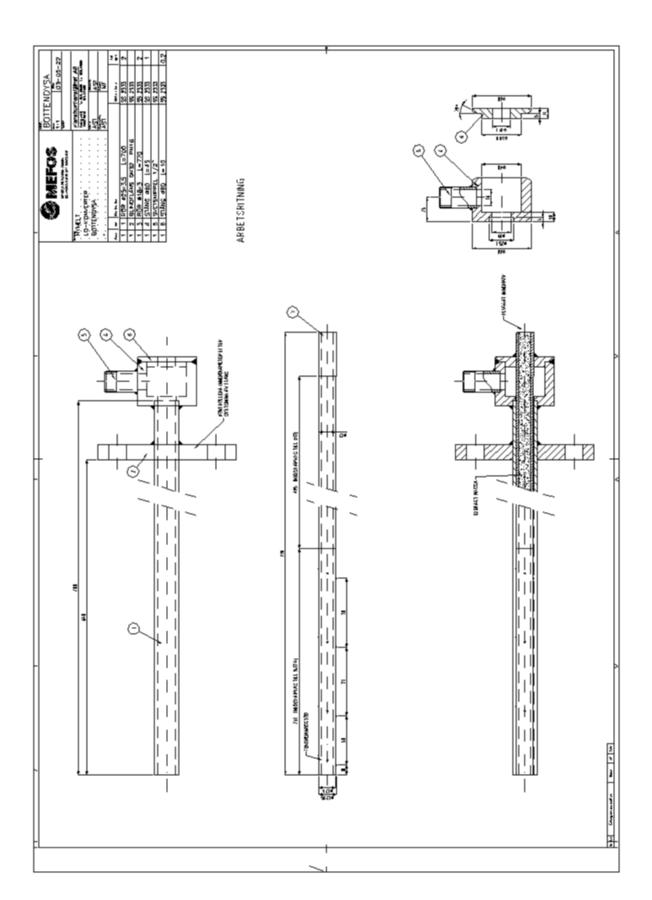


Figure 4 – Bottom tuyere

2.2.6 Gas and dust sampling lance for converter atmosphere

Process gas and dust was collected by a suction lance that could be lowered into the converter in parallel with the injection lance. The lance tip was made as an exchangeable ceramic probe of castable high alumina.

Technical data:Length3825Diameter76 mm

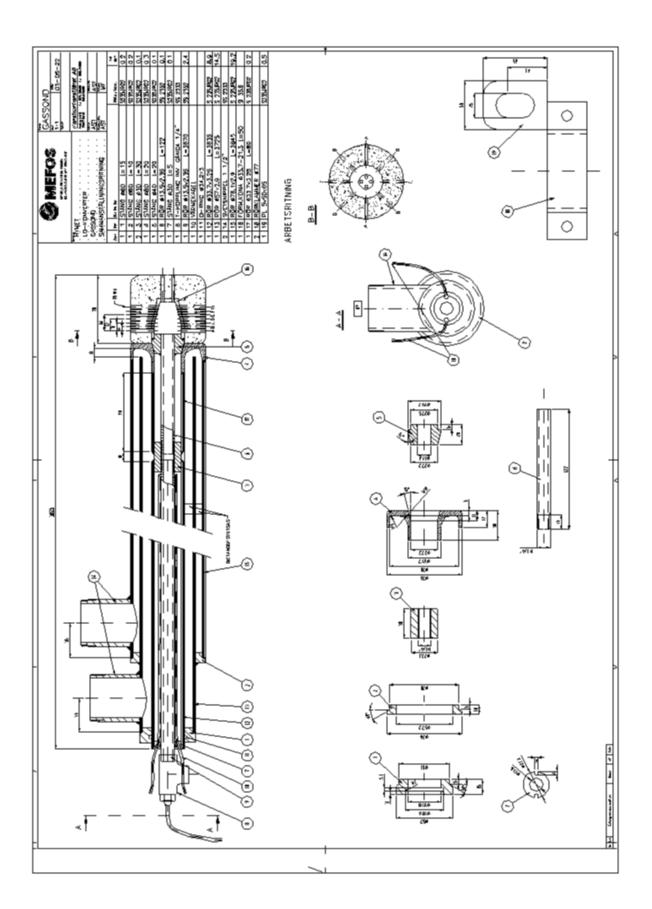


Figure 5 – Lance for gas sampling in converter

3 Material

3.1 Injectants

Materials used for the tests supplied by EnviRes.

3.1.1 Illinois #6

C H N Cl	71, 14% 4, 91% 1, 48% 0, 13%
S O	0, 15% 3, 48% 8, 26%
$AshSiO_2Al_2O_3CaOFe_2O_3$	10, 81% 50, 65% 20, 15% 4, 01% 16, 25%

3.1.2 Pet-Coke

С	86, 3%
Н	5,0%
Ν	1,0%

S 6, 5%

3.1.3 325 Aromatic extract

С	90, 31%
Η	6,99%
Ν	0, 24%
~	1 0 0 0 /

S 4,09%

3.2 Additives

Locally supplied material

3.2.1 Lime mix

The slag former used was a pre mix of burned lime and burned dolomite in 9 to 1 ratio.

Lime

Dolomite

3.2.2 Aluminium bars

Aluminium bars of \sim 5 kg were used as fuel additives for compensation of the heat balance. The addition was made before start of oxygen blowing.

96, 57%
0, 62%
1,41%
0,65%

3.2.3 Ferro silicon

Ferro silicon was used as fuel additives for compensation of the heat balance. The addition was made before start of oxygen blowing.

Si 92%

3.2.4 Ferro sulphur

Ferro sulphur was used in the last heat S1787 in order to drastically increase operational sulphur level in slag and metal. The test included purging of hydrogen through bottom the tuyere.

S	29, 18%
Р	0,01%
С	0,047%

3.2.5 Ferro vanadium

In order to indicate the material balance for vanadium the concentration of vanadium was increased in the last heat \$1787.

V	81, 88%
С	0,016%
S	0,023%
Si	1,23%
Al	0, 46%
Р	0,016%

4 Test procedure

4.1 Melting

Charging of pig iron was made the day before the test. The melting procedure followed standard routines for production of synthetic hot metal, ~4 % C and ~0, 2 % Si. Before tapping to a hot metal transfer ladle, the temperature was adjusted to ~1600 $^{\circ}$ C.

4.2 Decarburisation of hot metal

The converter operation started with oxygen blowing, 10 m³n/min, to ~1650 $^{\circ}$ C. During blowing, about 20 minutes, all silicon and some carbon from the metal were oxidised. To maintain a slag with reasonable fluid ability and melting temperature, the formed SiO₂ was neutralised by the double amount of lime mix. The lime mix consisted of 90% burned lime and 10% burned dolomite.

The oxygen lance and lime feed program was simplified to:

Time	Operation	Lance position above metal surface
0 min	Ignition	400 mm
1 min	Lime feed	800 mm
min 600 mm		

During blowing the metal temperatures were measured and samplings were made approximately every second minute. The samples taken were analysed for C and S by use of a Leco analyser.

4.3 Carburisation H₂ production

The oxygen blowing was followed by carburisation and H_2 production. The operation was made by top lance injection, ~10 kg/min, at a distance of about 400 mm above the metal surface. Temperature measurements and sampling were made as for oxygen blowing. The operation was stopped when desired carbon content was achieved or if the metal temperature became too low.

During blowing a probe for gas sampling was lowered into the converter, 700 mm above metal surface. The sampled gas was analysed for CO, CO₂, H₂, H₂S, COS, and CH₄. Alternatively the sampled gas was collected in a liquid for later Hg analysis. A filter, placed back of the lance, was changed after each blowing period and the collected dust was saved for later analysis. Dust sampling was also made by isokinetic suction from combusted gas in the off gas duct.

After the injection period the converter was tilted and the slag and metal could be inspected and sampled. The metal samples were of spectrometer quality for full chemical determination. If necessary some slag was skimmed of.

4.4 De-carburisation CO production

The procedure for de-carburisation followed mainly the routines for classical BOF blowing. Compensation for heat losses were made by addition of additional fuel, aluminium bars and FeSi before blowing start. To maintain reasonable slag properties the formed Al_2O_3 and SiO_2 was compensated by lime mix.

The procedure was followed by a new injection period or by tapping in case of the last trial for the day.

4.5 Tapping

Before tapping the metal temperature was adjusted to about 120°C super heat. This was made by oxygen blowing in combination with scrap or aluminium addition. After slag skimming, tapping was made into a steel ladle or hot metal ladle and cast into sand beds. The following day slag and metal was separated and weights were recorded.

5 Heat notes

5.1 HyMelt 1

Un-alloyed pig iron was melted in the EAF, tapping and charging of 5500 kg of hot metal was made without only small disturbances.

Heat ID S1782

Coal was selected for injection.

Totally five period of injection were made and initial problems were detected on:

- Level control of the lances had a poor function because of mechanical interference between cooling rubber tubes and malfunction of chain for the oxygen lance
- Sub-lance system had to be tuned for dipping time and sampling level
- O₂ flow-rate control and routines for the MEFCON system had to be tuned
- The dust load indicated that the complete system for process gas had to be cleaned after each injection period.
- The temperature drop during injection was unexpected high about 10 °C
- Low temperature injection <1400°C gave "unreacted coke" on top of the slag

Tapping and casting could be made without any problems.

New routines for data collection and conducting the test must be made for the coming day.

5.2 HyMelt 2

Heat ID S1783

Operation could be performed under good control. Totally five periods of coal injection and one period of coke were made.

- The "unreacted coke" on top of the slag could almost be completely avoided by operation at higher temperatures, melt temperature was maintained above 1400°C.
- The poor heat balance was managed by aluminium addition and by avoiding tilting of the converter between oxygen blowing and injection.
- 4 periods of process gas sampling was possible.
 - 2 gas analysis coal injection
 - 1 Hg-sampling coal injection
 - 1 gas analyse coke injection
- From the start of gas suction the pressure drop increased giving a continuously increased amount of leakage air to the analysers.

- Sculling, narrowing the converter mouth gave some problems with lance movements
- The last injection was made with coke giving strong indications of an improved carbon yield and an improved heat balance.
- The feeding rate was reduced during coke injection. It was later found that some rubber material was blocking the outlet.

5.3 HyMelt 3

Heat ID S1784

Pet-coke was used for all injections periods. Totally five periods was made and gas suction of process gas was possible for all periods. An overall improved heat balance gave drastically reduced aluminium addition. This in combination with no ash in the material gave a low slag trough put and a stiff and dry slag. It was understood that the slag has been saturated with MgO from refractory wear during the day.

Moderate feed rate 10 kg/min gave in first two injection periods excellent result. Attempt to increase the rate up to 20 kg/min was only possible by reducing the carrying gas from $0,55 \text{ m}^3\text{n/min}$ to $0,35 \text{ m}^3\text{n/min}$ and by increasing the nozzle outlet diameter from 7,0 to 7,5 mm at ~12:00. Both these action caused a lower impulse of material jet to the bath. It was later found that coal was by mistake charged to the dispenser during the day.

- Large amount of coke was trapped in the slag giving flames spontaneously or when the slag was skimmed.
- The dry slag gave problems to measure in blow temperatures.
- The heat was tapped when water leakage occurred on the oxygen lance.
- Remaining problem with increased amount of leakage air during sampling of process gas. The analysed components must be corrected by use of an oxygen balance. Argon content is therefore included from this day in the database. Clogging is mainly caused by dust in the filter in top of the lance and in the ceramic head.
- The standard hydrogen analyser was closed before the start of the tests.
- Recalibration of MS hydrogen was made after that calibration gas of 100% responded only 85%. Can be seen in the data base.

5.4 HyMelt 4

Heat ID S1785

Pet-coke injection was made for 5 periods, gas analysis was possible for all. During the day the confusion between coal and pet-coke was detected.

A new slag was tested, the purpose was to achieve good fluid ability down to 1400°C. The slag was made of oxides from FeSi98 and aluminium bars fluxed with lime and dolomite mix. The result was acceptable and slag skimming could be made after the injections.

The injection nozzle was drilled to 8 mm to allow higher feeding rate up to 20 kg/min. However at this rate the transport gas had to be lowered to 0, 3 m^3n/min giving a lower impulse from the gas/material jet into the melt. This was during the day compensated with higher gas flows 0, 7 m^3n/min and a lower material flow rate.

The operational lance distance was measured with a steel bar placed on the material lance nozzle. The result confirmed calibration 400 mm indicated metal up to 280 mm. The difference is caused by unstable metal surface from bottom stirring.

Preliminary mass balance shows low carbon yield to the melt, the losses to combusted gas was typically 3 kg/min and coke in the slag was found in the same magnitude. High CH_4 % was also reported from process gas analysis.

Sculling on the oxygen lance was detected for the first time and had to be cleaned several times. It was also found that the nozzle of the material lance was clogged in home position during oxygen blowing. It was easily detected when carbon injection was started with the material plume in the slit between converter mouth and the hood. This small scull was removed before all tests except from the first injection which was operated with a poor plume.

The moderate carbon yield can be caused by:

- Particles not solute directly in the jet-metal interface will float up on slag surface giving only small possibilities to contact with metal. With a stiffer slag it is more likely that metal surface and coal particles in direct contact.
- Lower jet impulse from the larger nozzle diameter.

Later note: material confusion caused the main problems with C-yield.

5.5 HyMelt 5

Heat ID S1786

Injection of oil was made for the first 4 periods, the remaining day coal was injected for 3 periods. Oil seems to be a more difficult source than solids. Large amount of CO_2 was detected in the combusted gas indicating severe soot formation.

The coal tests showed similar results as previous heats, moderate carbon yield, tendency for build up of coal on top of slag and a weak heat balance. The lance was made with a 7 mm nozzle.

At 14:30 it was discovered that one of the coke bags was confused with coal. Until then coal has been injected as coke. This can explain the problems to repeat the good results achieved earlier with coke. Experiments made at HyMelt 4 must be rechecked.

Good gas suction of process gas for analyse. The last two periods was used for mercury sampling.

5.6 HyMelt 6

Heat ID S1787

Totally 6 injection periods with pet-coke were made at controlled conditions. Tests at higher V and S levels. Hydrogen purging at high sulphur level was performed

6 Result

The complete data base and the individual test periods have been distributed separately. Below is supplementary data presented as graphs. Detailed information on individual test periods can be found in Appendix 1.

6.1 Process gas analysis

In Appendix 2 is an overview of measured components in the process gas presented. Detailed graphs for individual injection periods are presented separately and comments made do also refer to those.

A general difficulty was clogging of the sampling probe giving an increased amount of leakage air in the gas sample during the measuring periods. For the evaluation the recorded values have been balanced by use of the measured oxygen for compensating the amount of air dilution

Isolated peak values are related to calibration and frequent peaks especially on CO are caused from partial combustion of sub-lance probes.

6.1.1 CO

The recordings show significant difference between the injectants.

- Coal gives stabile CO concentration in the range of 20 30%.
- Coke gives a decreasing CO concentration during injection starting around 20% down to 10%.
- Oil shows the same behaviour as coke but on a lower CO concentration down below 5%.

The measurements seem to mirror the oxygen content in the material and that we have to consider declining reduction of oxides in the slag during injection.

6.1.2 CO₂

It is not expected to have significant amount of CO_2 in the process gas. The presence can be due to leakage air into the converter atmosphere or to leakage in the sampled volume. The readings are generally below 5% and frequently below 1%.

6.1.3 H₂

The H_2 measurement has a long response time, up to 5 minutes when calibration with a fixed concentration and it is accordingly to be expected an increased reading during the injection period. Coal and coke generates a gas of 50% to 60% H_2 , occasionally 10% higher or lower, while oil shows about 70% H_2 .

6.1.4 CH₄

Coal and coke give gases of 5 to 7 % CH₄ while oil gives 8 to 12%.

The accuracy is not perfect since the component gives a stronger signal than recommended for optimal performance.

6.1.5 H₂S

The process gas has a content of 0,2 to 0,4 % H_2S independent of injectant. At higher sulphur level in metal the H_2S content in the gas is somewhat higher, 0,5%. (0,8 to 1,0 %S compared to normal level 0,1 to 0,3 %S)

It has not been shown that purging with H₂ will effect the H₂S concentration.

6.1.6 COS

The graphs shown in the Appendix 2 are based on the spectrometer data base, since the records in the Lab View system do not mach. Unexpected high values have been measured, frequently more than 500 ppm, more typical recordings are in the range 100 to 300 ppm.

The data base in the spectrometer operates with complete different format and it is preferred if all future measurements are transferred to the Lab View system.

6.1.7 O₂

No oxygen is expected to be in the gas, the presence is most likely due to leakage into the cooled gas sample.

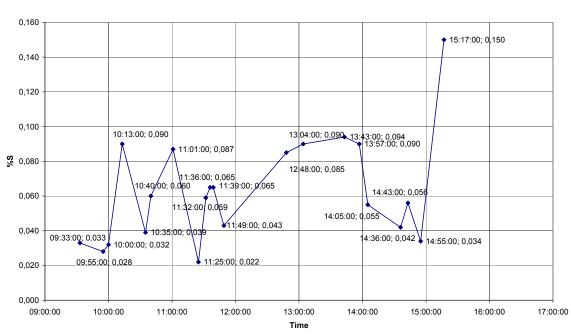
6.2 Metal analysis

Below are the metal analysis presented for each element of interest. The analyses are generally believed to be good enough for the coming campaigns. However, the in-blow

samples of spectrometer quality are few and the sampling procedure must be improved. Carbon and sulphur were analysed on the Leco system which operates with samples of lower quality.

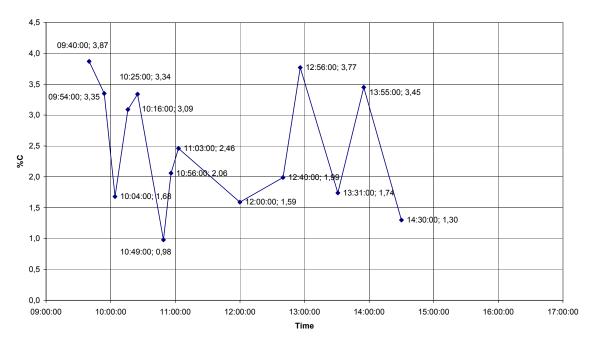
Complete analysis of metal and slag is shown in Appendix 3

6.2.1 Carbon S1783



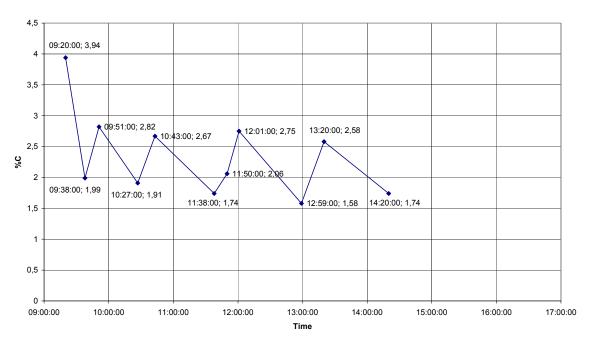
S1783 %S in metal

S1784 %C in metal

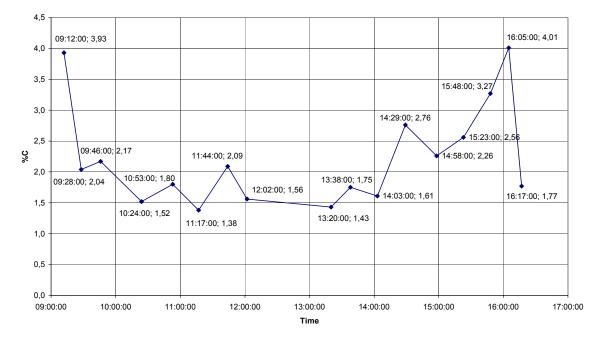


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S1785 %C in metal



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S1786 %C in metal
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-AI p29 -

S1787 %C in metal

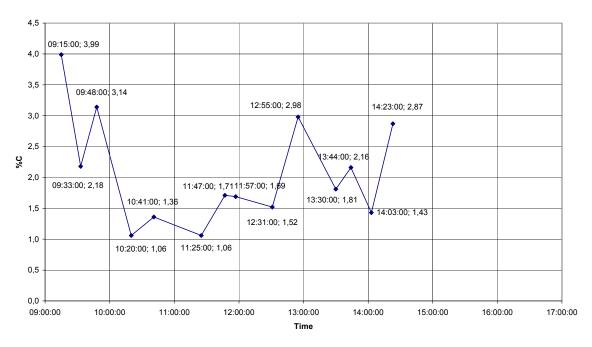
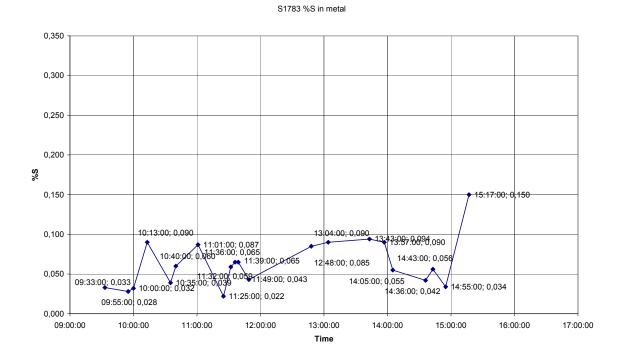
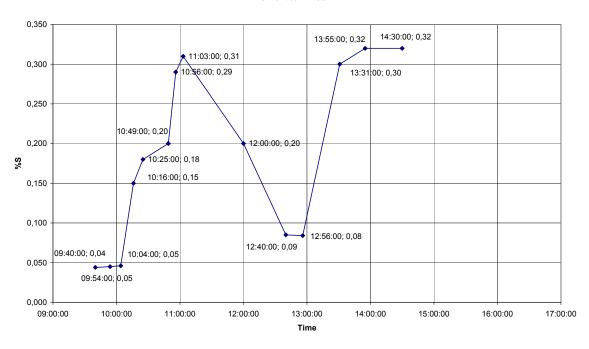


Figure 6 – C analysis in liquid metal HyMelt 2 to 6



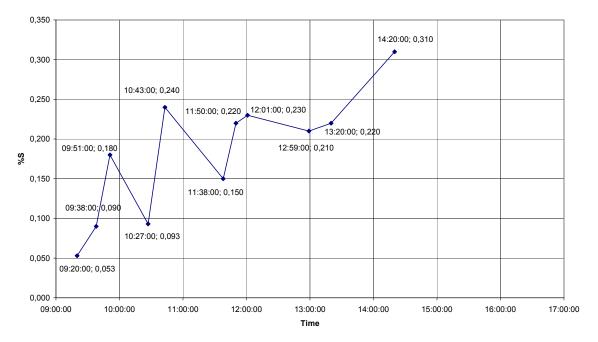
6.2.2 Sulphur

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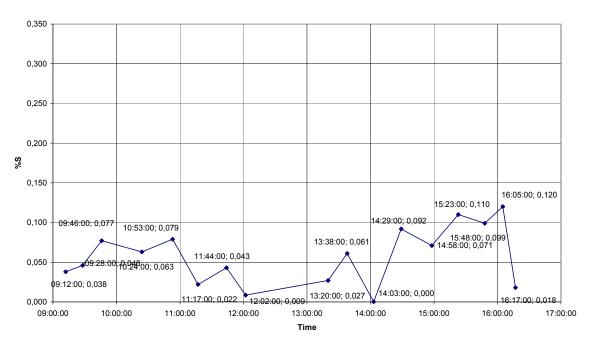




S1785 %S in metal



S1786 %S in metal





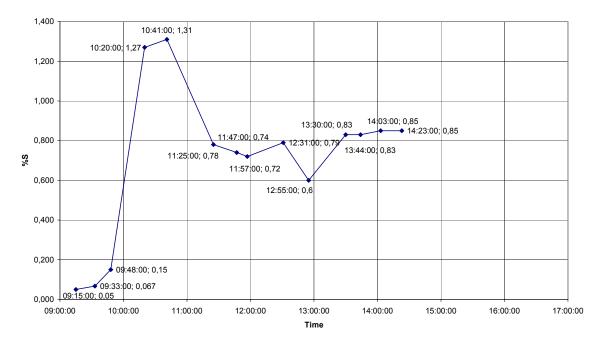
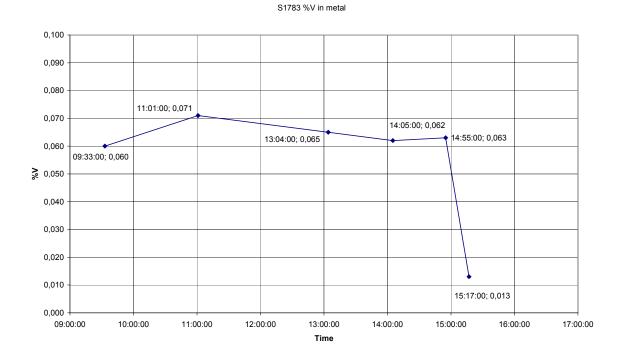


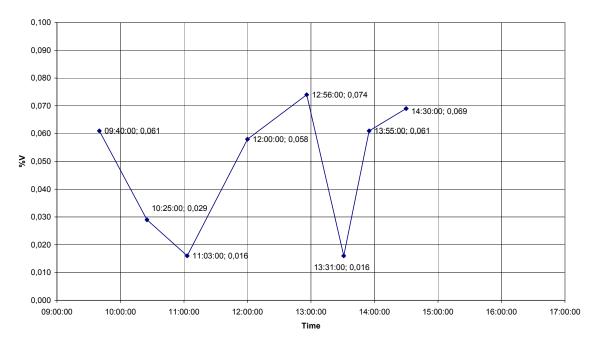
Figure 7 – S analysis in liquid metal HyMelt 2 to 6

-AI p31 -



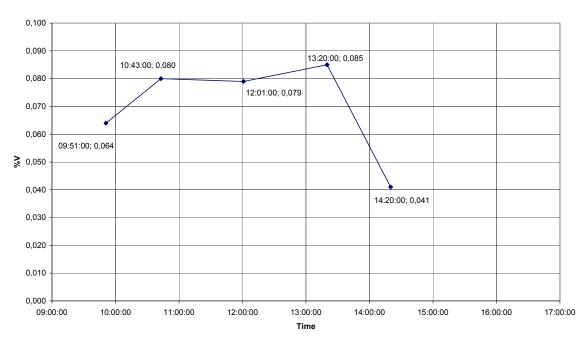
6.2.3 Vanadium

S1784 %V in metal

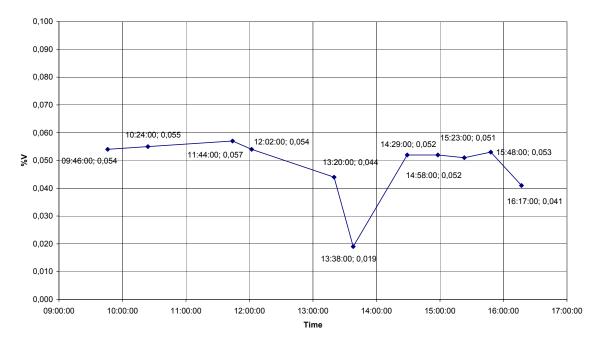


-AI p33 -





S1786 %V in metal



-AI p34 -



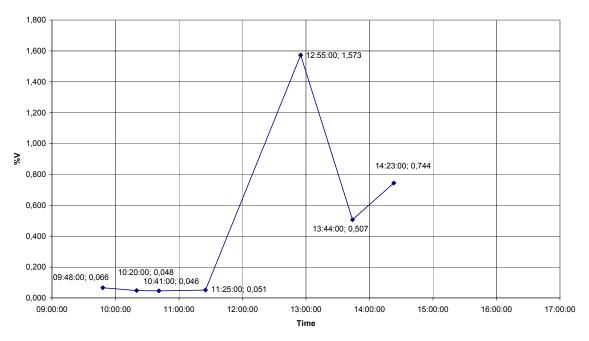
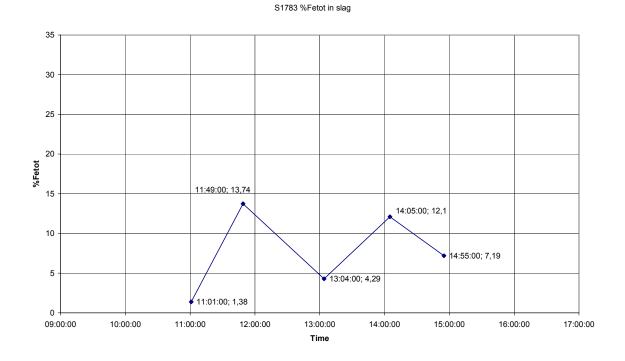


Figure 8 - V analysis in liquid metal HyMelt 2 to 6

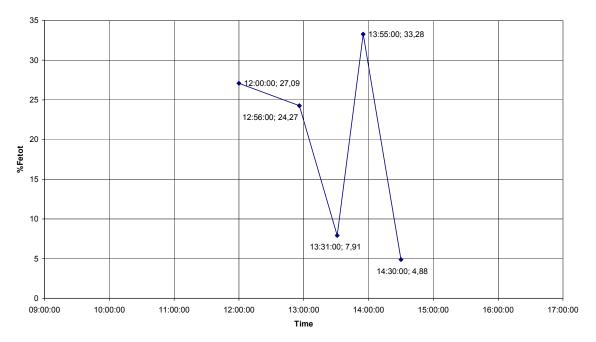
6.3 Slag analysis

Slag samples are only possible to collect from tilted converter. The method can be used in coming campaigns.



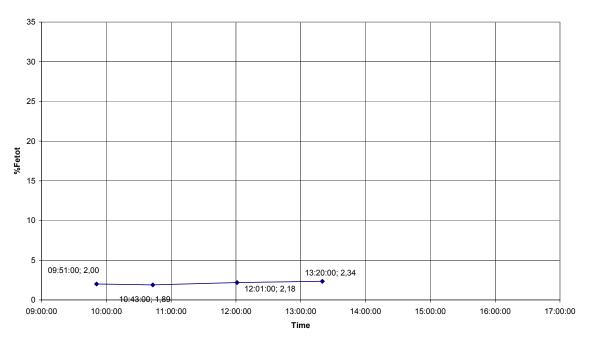
6.3.1 Iron oxides



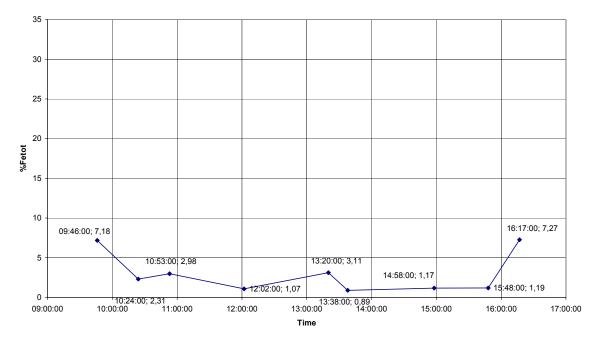


-AI p36 -









-AI p37 -



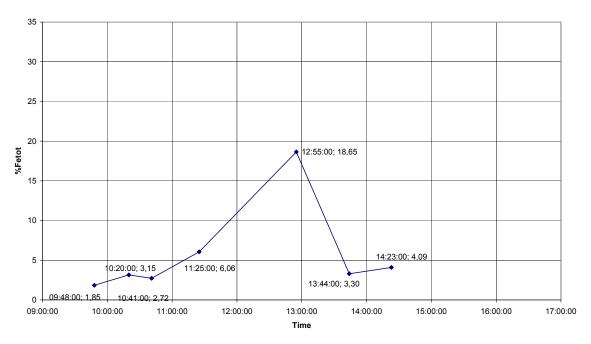
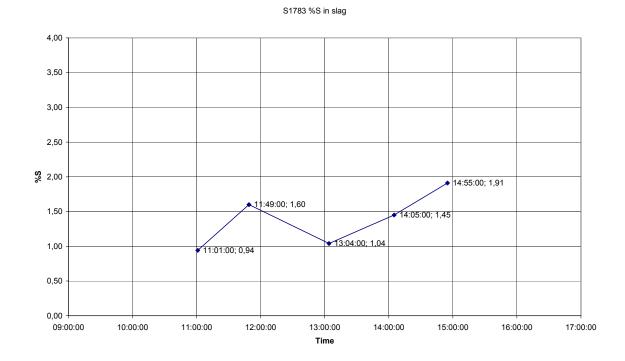
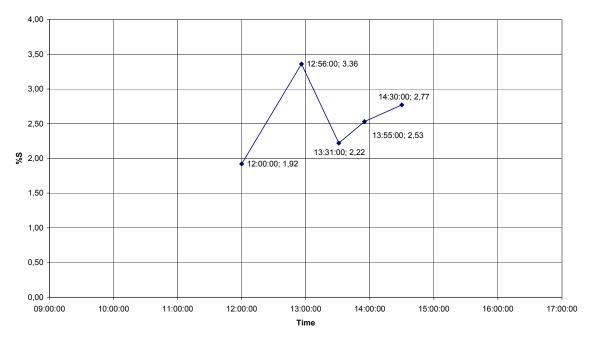


Figure $9 - Fe_{tot}$ in slag HyMelt 2 to 6

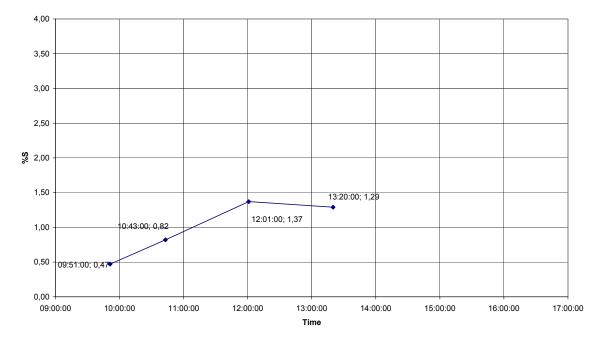


6.3.2 Sulphur



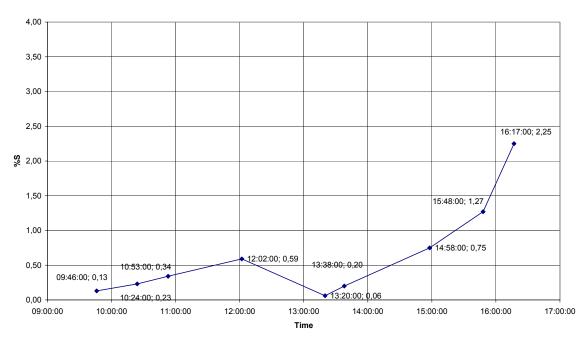


S1785 %S in slag



-AI p39 -







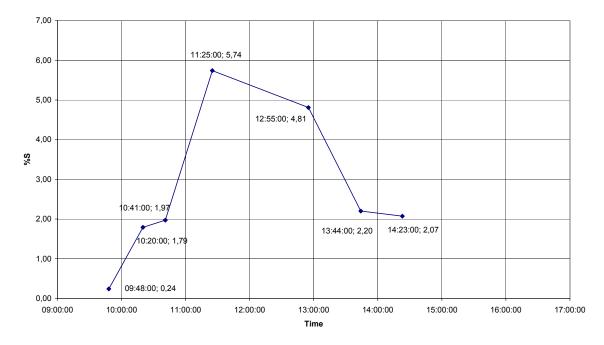
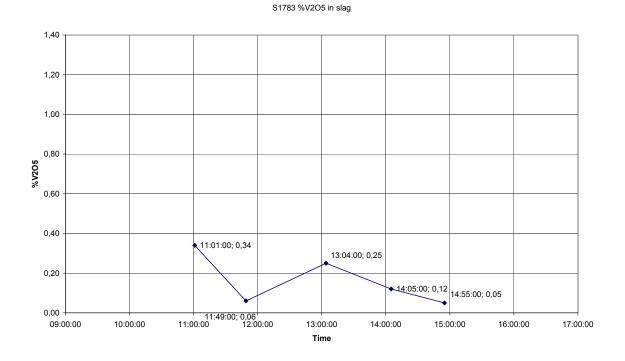
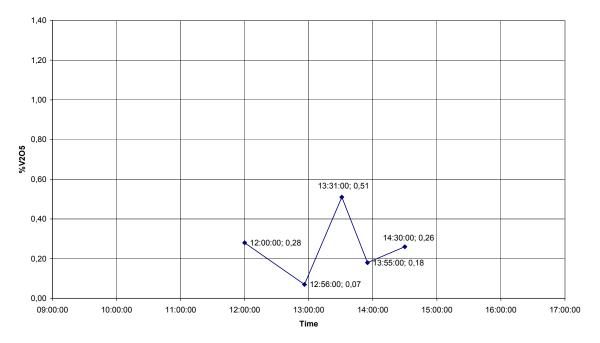


Figure 10 - S in slag HyMelt 2 to 6

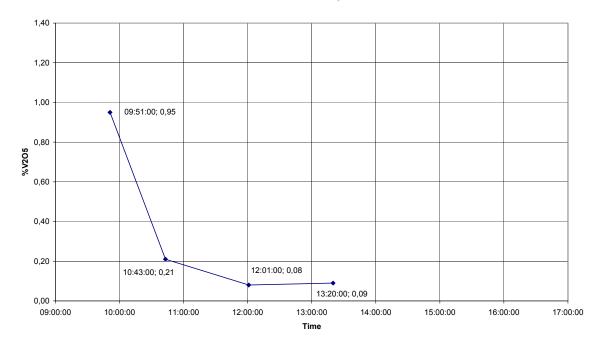


6.3.3 Vanadium oxides

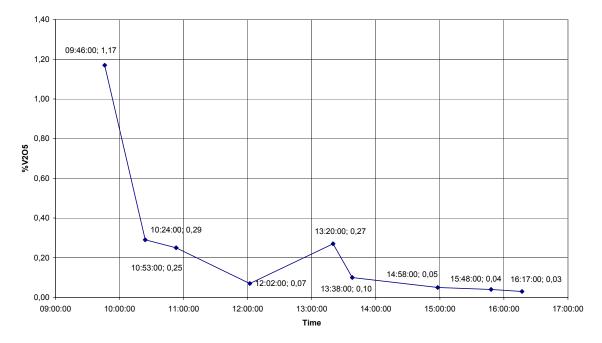




S1785 %V2O5 in slag



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S1786 %V2O5 in slag
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-AI p41 -

-AI p42 -



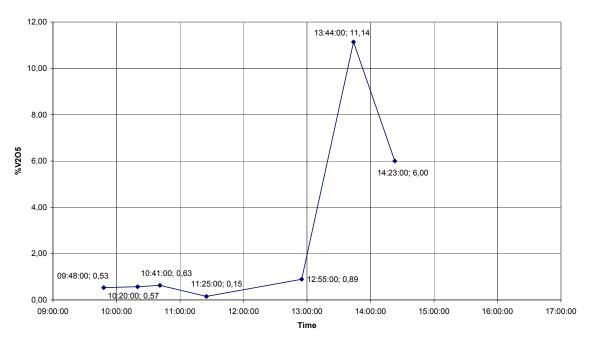
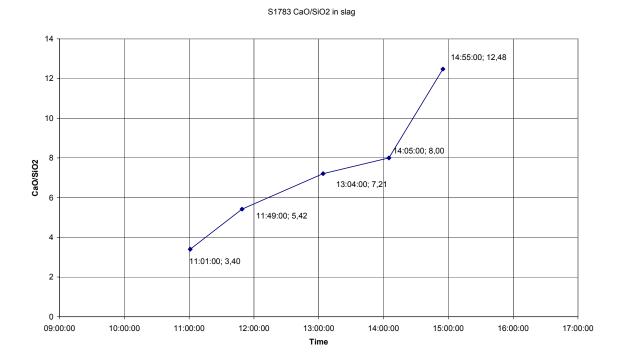
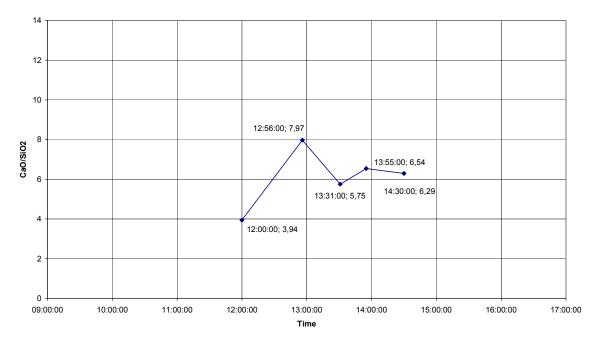


Figure 11 – V-oxides in slag HyMelt 2 to 6

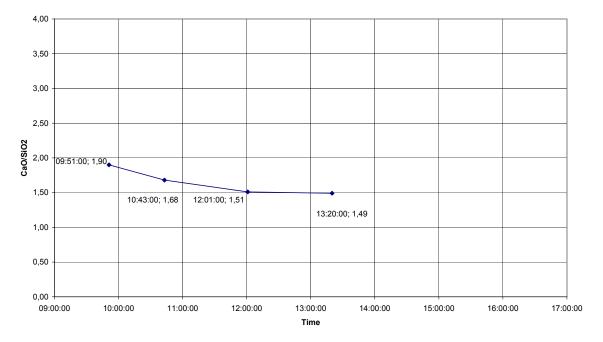
6.3.4 Bas CaO/SiO₂







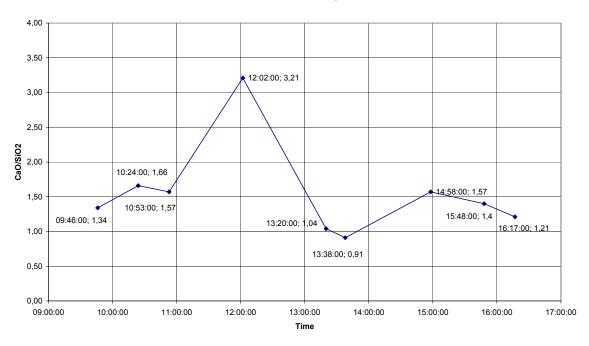
S1785 CaO/SiO2 in slag



-AI p43 -

-AI p44 -

S1786 CaO/SiO2 in slag



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S1787 CaO/SiO2 in slag
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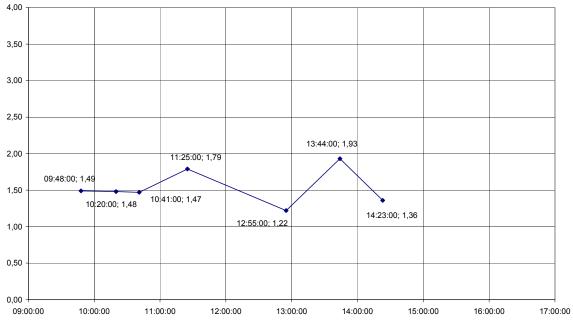
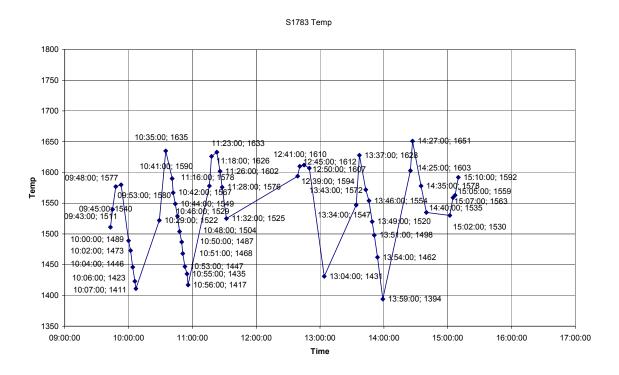


Figure $12 - CaO/SiO_2$ ratio in slag HyMelt 2 to 6

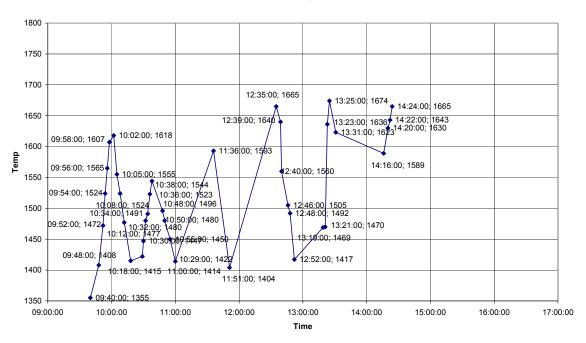
6.4 Temperatures

The metal temperatures refer either from sub-lance measurements or manual lance measurements. The system and code for evaluation of the EMK must be improved in the Lab View system, presented values are manually evaluated.

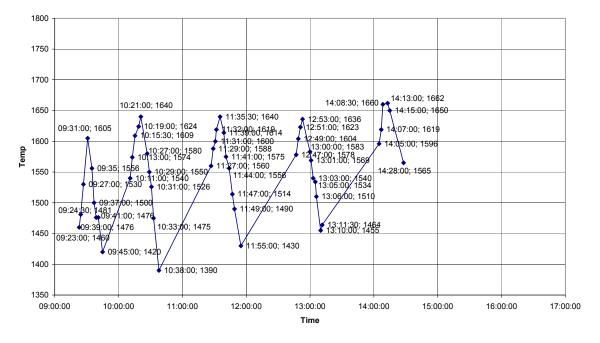


-AI p46 -



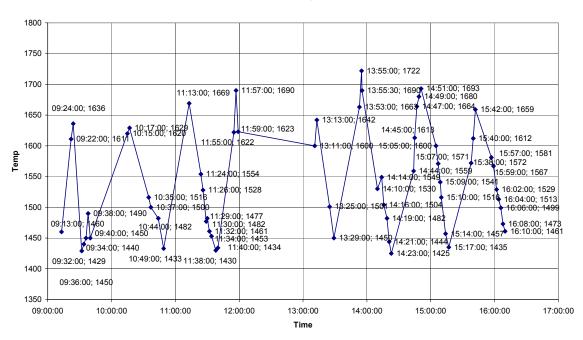






-AI p47 -







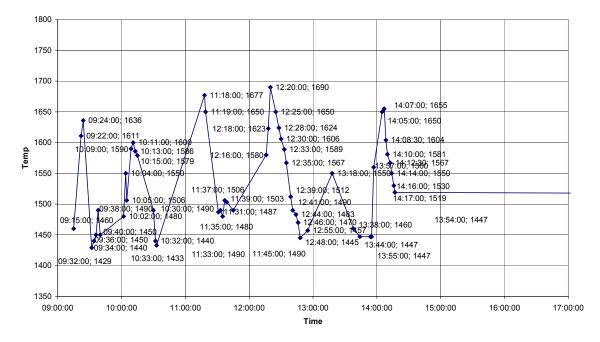


Figure 13 – Temperature in liquid metal HyMelt 2 to 6

6.5 Carbon yield to metal

The carbon yield has been calculated for all injection periods. The calculation is based on the first and last metal analysis in the period and the injected amount of carbon during the time between sampling. Details are shown in Appendix 4.

The best results were archived for pet-coke at moderate feeding rate. A rather strong dependence between yield and feeding rate can be seen. The expected relation between nozzle diameter and yield can not be detected.

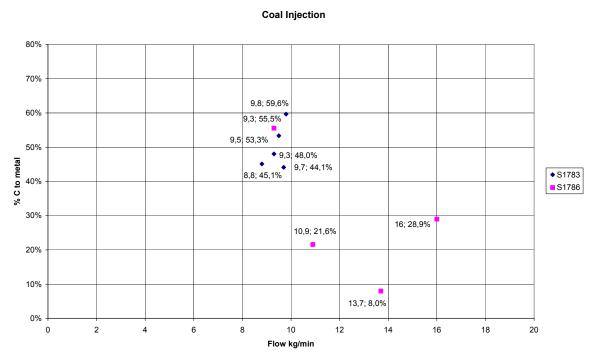
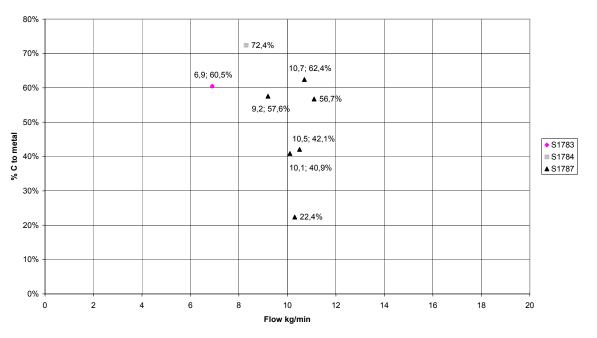


Figure 14 - C yield for coal injection

-AI p49 -



P-Coke Injection

Figure 15 - C yield for coke injection

Mix Injection

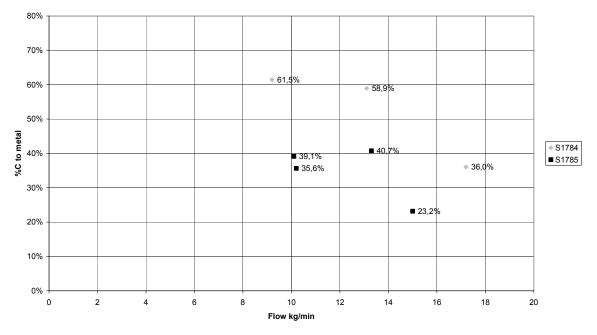


Figure 16 - C yield for coal/coke mix injection