



Process Mass Spectrometry and the Steel Industry

Aspec. Application Note

Process Mass Spectrometry and the Steel Industry



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- Principles of Mass Spectrometer
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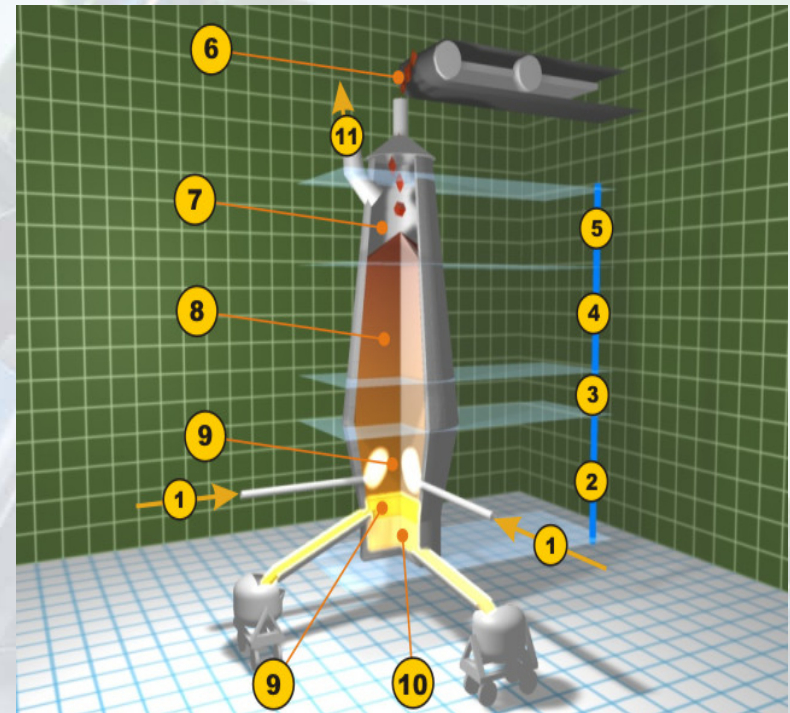
Blast Furnace Example

A typical blast furnace is used to produce pig iron. Fuel: Fuel ore, flux (limestone) are continuously fed to the top of the furnace. Whilst often Oxygen enriched air is fed into the bottom. Product moves slowly down through the furnace and a series of chemical reactions takes place resulting in a carefully controlled raw product. Throughout the iron making process waste gases are produced. Measuring and monitoring these gases precisely using Mass Spectrometry allows the operator to control the process and efficiency of the furnace.

Benefits of using Mass Spectrometer analysis.

- Maximise Iron Production
- Minimise Coke usage
- Consistent raw product
- Safer operation

Gas Component	Typical Aprox Range %
Nitrogen	10-80%
Carbon Monoxide	5-70%
Carbon Dioxide	1-50%
Oxygen	0-25%
Hydrogen	0-5%
Argon	0-1%



- 1) Hot Blast
- 2) Melt Zone
- 3) Ferrous Oxide Melting Zone
- 4) Ferric Oxide Reduction Zone
- 5) Pre-heating zone
- 6) Feed: Ore, limestone, coke
- 7) Exhaust Gas
- 8) Column of ore, coke and limestone
- 9) Slag removal
- 10) Moulton Pig Iron
- 11) Collection of waste gases

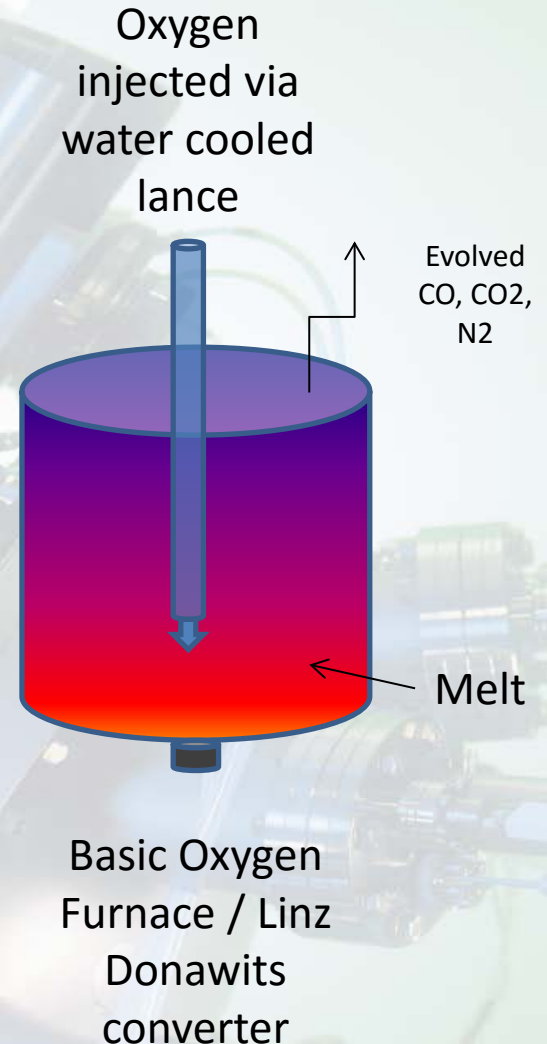
MS use in Primary Steel Production

Typically the MS can be easily used to measure CO, CO₂, N₂, O₂ during this process in Basic Oxygen and Linz Donawitz converters.

Raw molten steel coming from the Blast Furnace may require the carbon content reducing from about 5% to as low as 0.2%.

This is often carried out by injecting Oxygen through a water cooled lance that is inserted directly into the melt and may reach 3,000 deg C. The action of the injected Oxygen causes oxidation of the carbon held in the melt and forms Carbon monoxide and Carbon dioxide as an off-gas. The Oxygen, Carbon monoxide and Carbon dioxide can all be measured WRT time and a typical carbon reduction cycle may last about 20 minutes.

Often fluxes are added to the de-carbonised steel to react with other contaminants and reduce their content along with the trapped Nitrogen.



Bulk Generated Plant Gas

Many large Steel plants require large amounts of bulk gas. These are primarily Nitrogen, Oxygen and Argon and are separated from Air. Often third party companies operate bulk gas Cryogenic Separators that provide this bulk production gas.

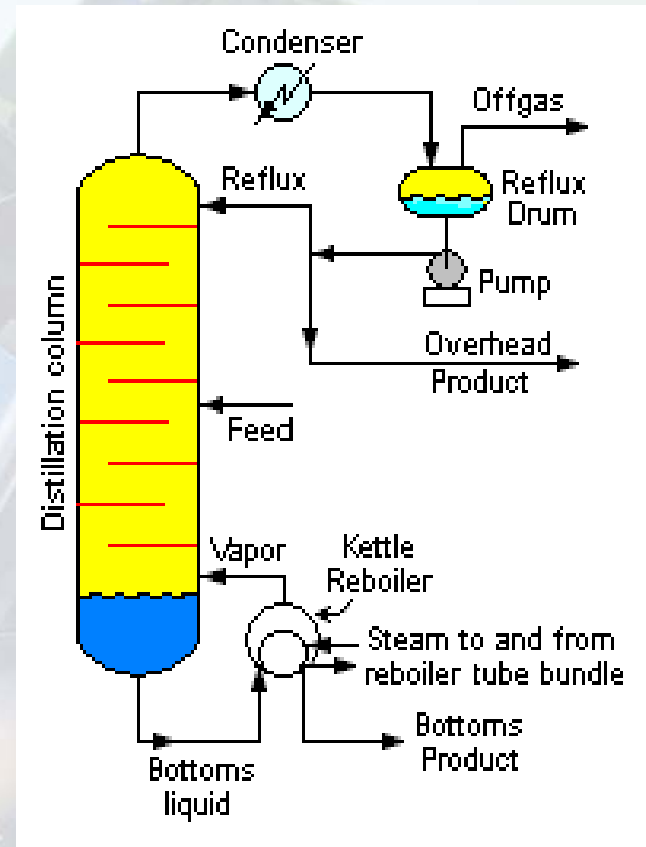
Air is pumped into a molecular sieve and the water, Carbon dioxide and oxides of Nitrogen are removed.

The dry Nitrogen, Argon and Carbon dioxide are then passed into a distillation column and separated into their individual components.

There is a use for MS during all or part of this process gas separating and purification process as the MS can not only be used for process monitoring but may also be used as a continuous on-line monitor for contaminants within each of the three separated bulk gases.

For example, the MS would be used to measure for the presence of Nitrogen in the pure Oxygen separation.

The use of Argon as a stir gas is preferred when trying to produce low Nitrogen based sheet metals for say car bodywork.



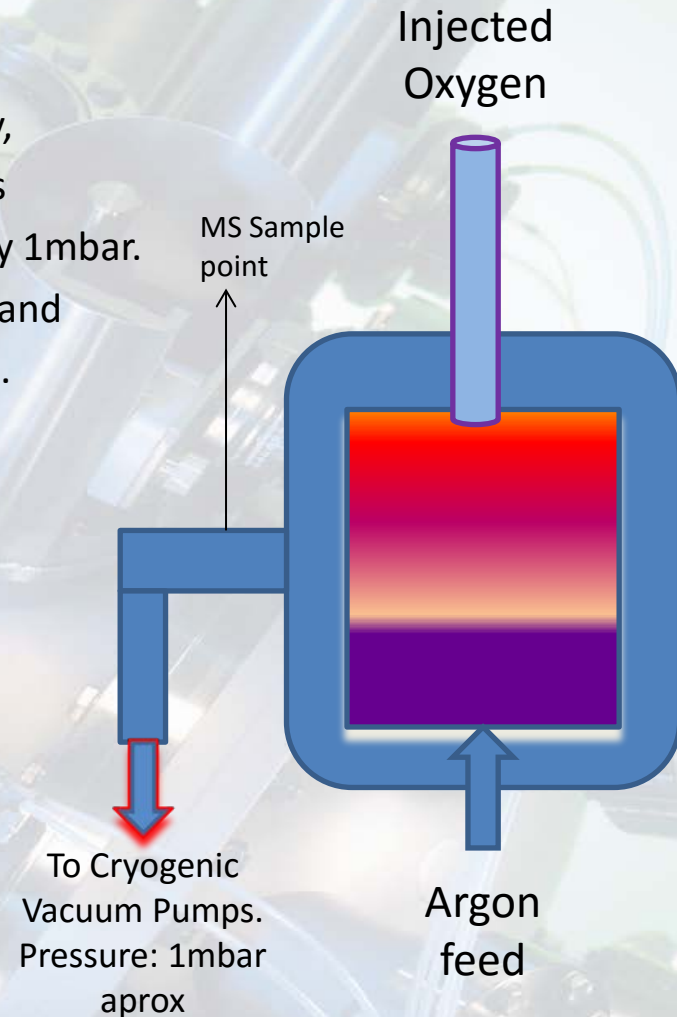
Secondary Steel Production

Here is an example of **Vacuum Oxygen Decarburisation** process.

This method is used for the creation of high quality, low carbon steel such as Stainless steel. The melt is secured and exposed to a vacuum of approximately 1mbar.

At the same time Oxygen is blown across the melt and Argon is purged through the melt from the bottom. This has the effect of removing the impurities. The Steel melt is actually been degassed.

The entire process is monitored WRT time using the MS. Helium gas may also be used as a tracer And again this volume measured by the MS.



Electric Arc Furnace

The Electric Arc furnace as its name suggests utilises the power, typically 100mw, of electric current and voltage to heat the contents of the furnace and create a melt.

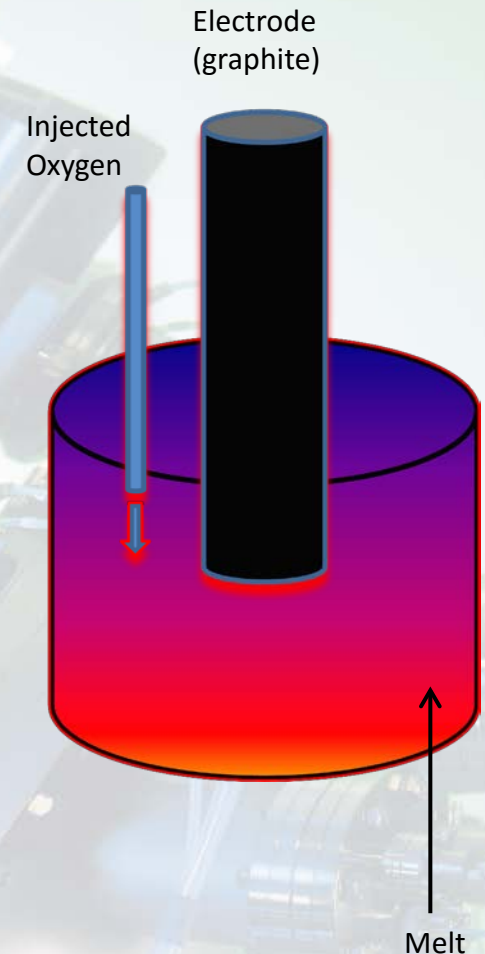
Typically the Electric arc furnace is charged with scrap. During the process Oxygen is added to increase the temperature and reduce the carbon content it also carries out the purification process along with the help of limestone etc that forms the slag and this absorbs the impurities.

MS again may be employed to measure the entire process which may give the following benefits:

Efficient Oxygen use.

Lowers electrical power consumption.

Improves operation safety



Process Fuel Gas

This is a complex area and in theory would benefit from a gas analyser that gives all the information required to deliver and predict the efficiency of the fuel gas burn during the process continuously.

Ideally, information such as a continuous Wobbe Index, Gas density, Calorific value, complete gas composition, gas volume etc would be preferable but compromises have to be made.

The MS is able to provide continuous fast analysis of gases such as CO, CO₂, H₂, CH₄, N₂, O₂, C₂H₆, C₃H₈, C₄H₁₀, C₆H₆.

This gas composition data combined with the Wobbe Index and flow/volume enables a good degree of control and forward prediction of the burn efficiency and air consumption.

Principles of the Mass Spectrometer

Sample gas is continuously injected into a the vacuum housing held at approximately 1×10^{-6} mbar.

When the sample gas molecule comes in contact with an electron emitted by a hot filament it becomes positively charged and forms an ion or a series of ions (cracking pattern) in predicted ratios.

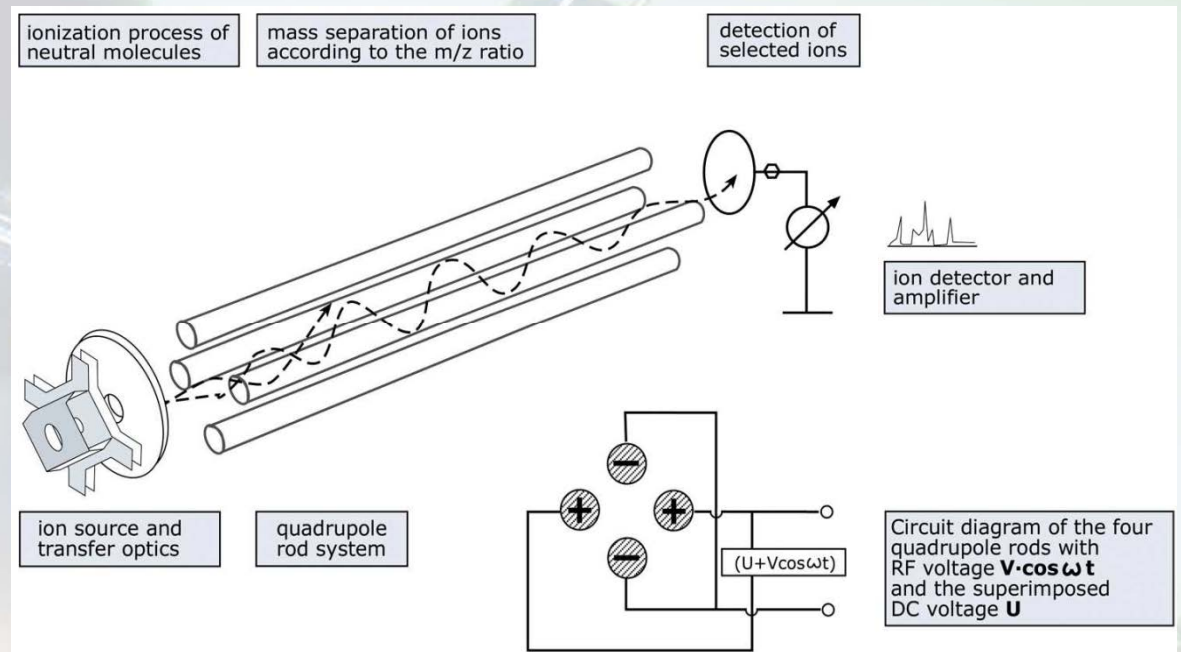
These ions are focused from the Ion Source via transfer optics towards the mass filter (quadrupole rod system). The Rod System consists of 4 accurately ground stainless Steel rods mounted very accurately into a set of ceramic carriers.

Once the ion enters the mass filter they are separated into their individual ion weights by the influence of both RF and DC voltages that are applied to apposing rods to create an electrostatic field.

Ions that leave the Mass Filter Rod Assembly strike a detector and produce an electric current.

It is an important principle that the Filtered Ion Current produced at the detector is directly proportional to the concentration of each individual gas species.

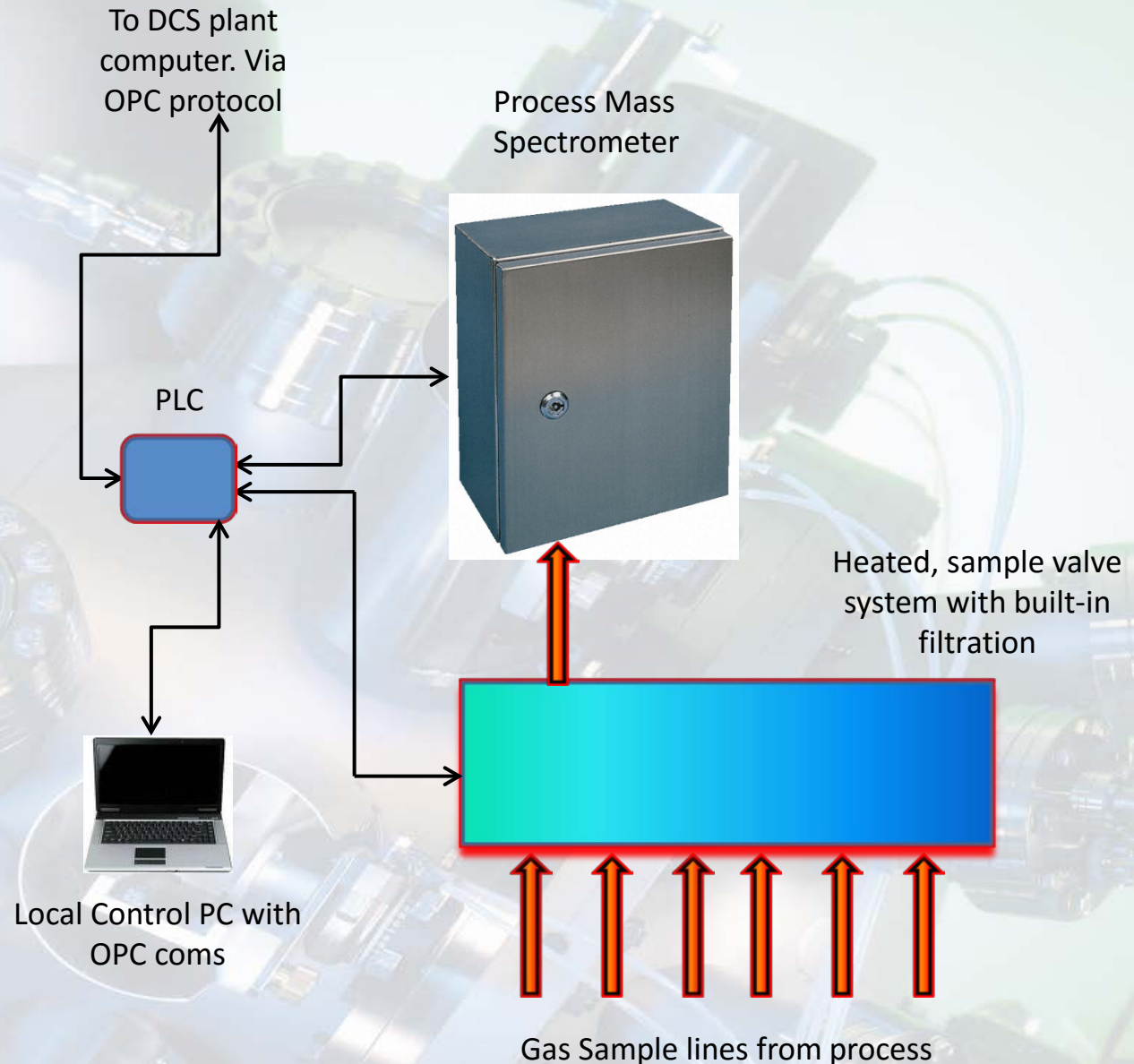
This whole Ionisation of the gas sample, transmission through the mass filter, detection and plotting on a computers Y axis takes far less less than 1ms making a Mass Spectrometer extremely fast.



Layout of Process Mass spectrometer

The entire analysis system communicates via OPC protocol that sits on the Local Control PC data system.

Many peripheral devices , in this case Process Mass Spectrometer, Heated sample valve system may be connected and OPC also supports many other input data streams such as Temperature, Flow etc. All data is visible to the main plant DCS at any time.



Mass Spectrometer Analysis

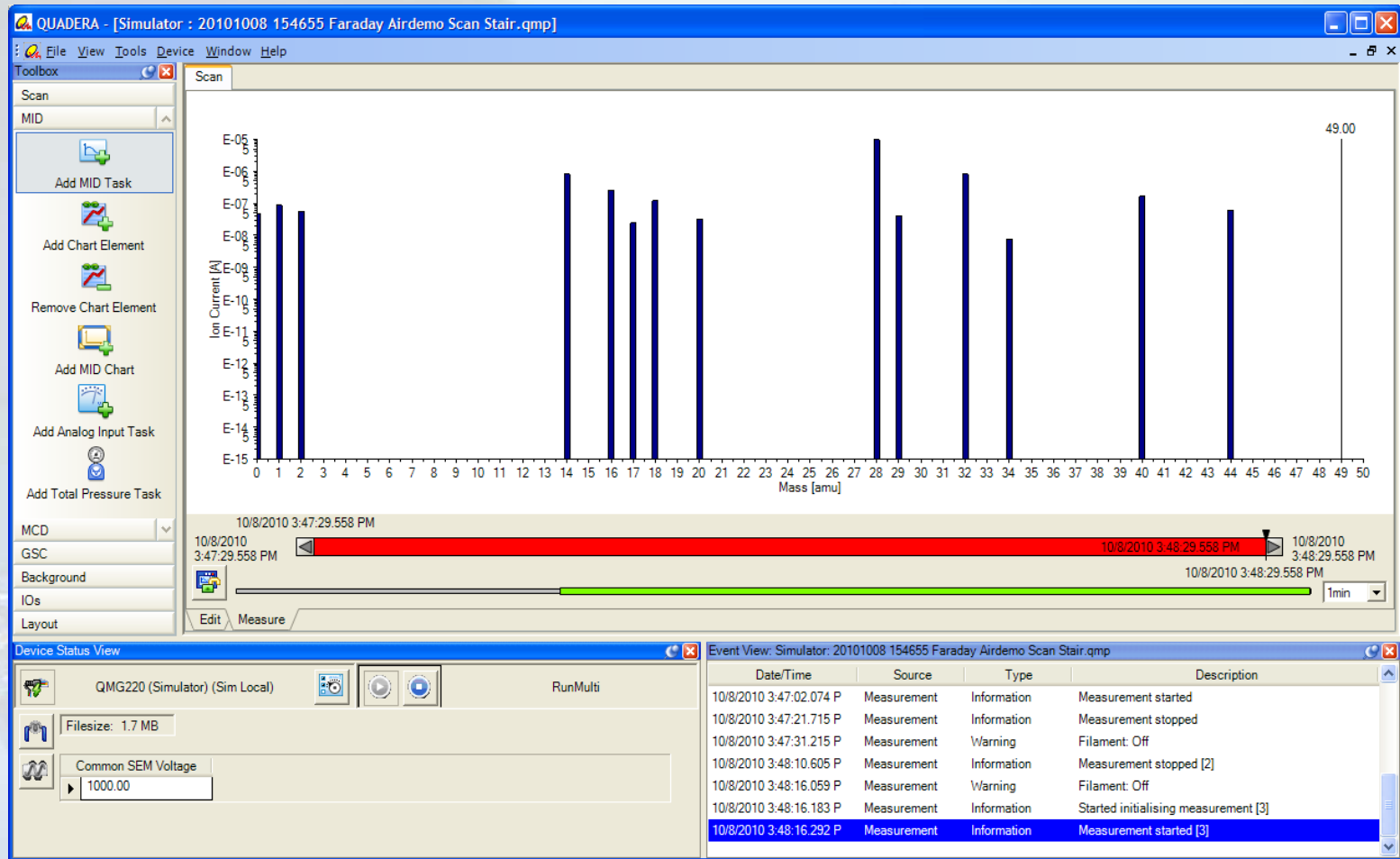
The Mass Spectrometer instrument is an extremely fast and flexible instrument and has the capability to measure up to 64 gas species at any one time.

The choice of gas species may be selected by the operator at any time or a standard matrix of gas species may be stored under a single analysis file and launched when required.

Here are 3 analysis mode examples that demonstrate the capability and power of the Mass Spectrometer instrument

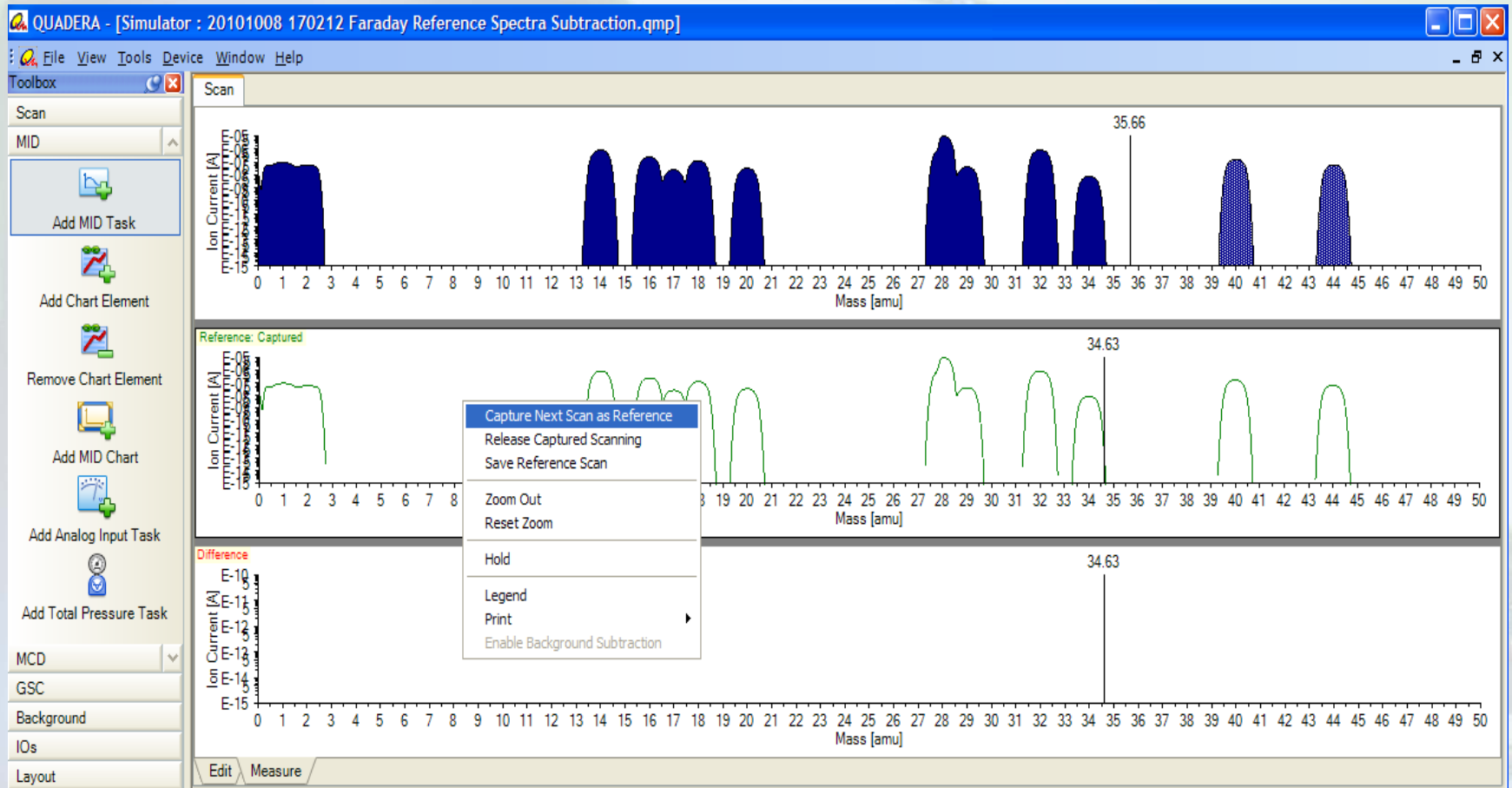
- Raw data **scan** mode.
- Background process subtraction (for looking at unknowns)
- Full Calibration data collection mode and data logging WRT time

Raw Gas/Mass scan Mode



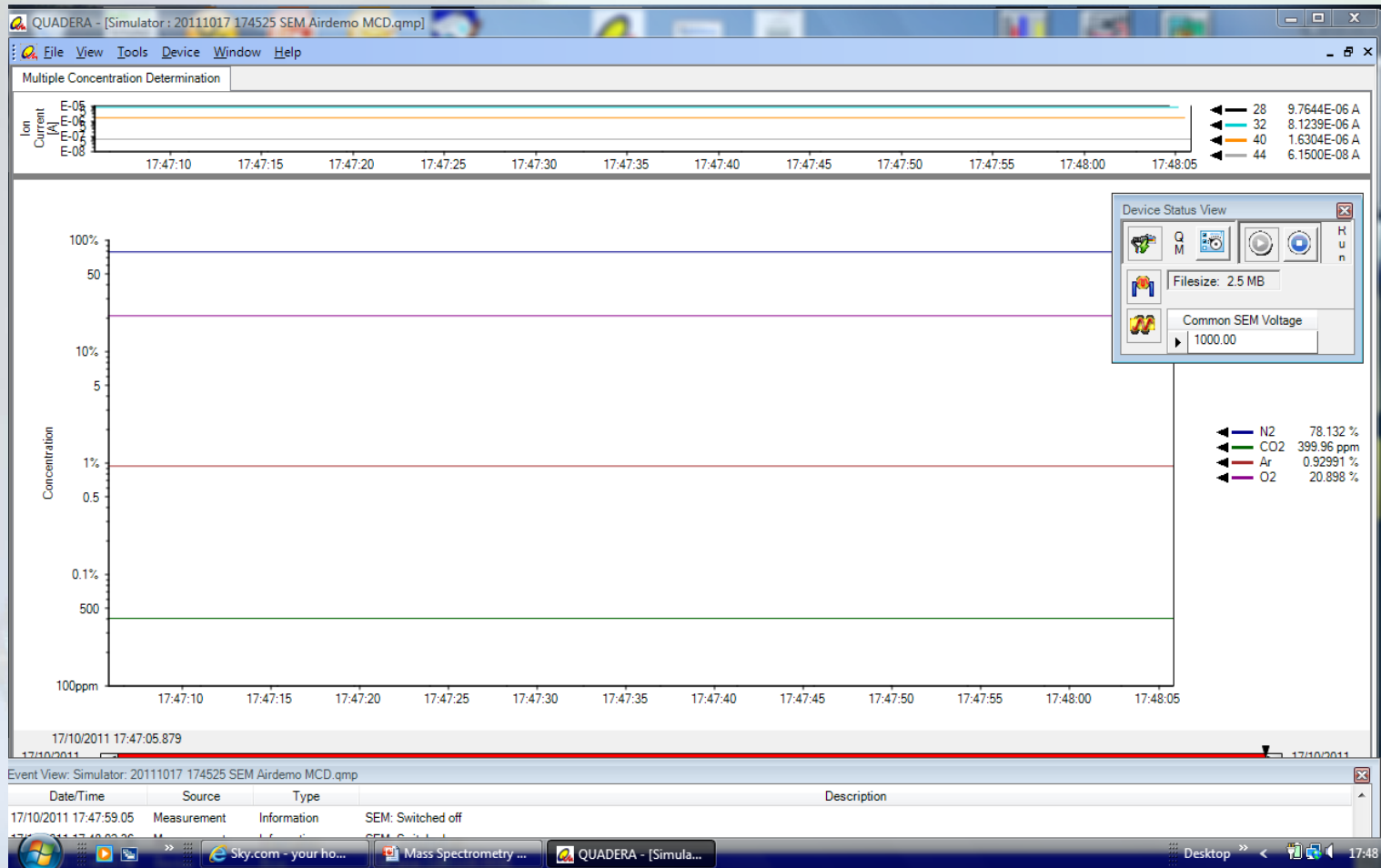
In this mode, you can “scan” the entire mass range of the instrument in less than 2 seconds and produce a complete spectra of any gas composition. The spectra will automatically show very low as well as very high concentrations all on one single spectra. Each bar of the graph above represents a different gas or isotope of a gas. The higher the signal, the more gas there is. This mode enables the user to quickly see if the gas composition is accurate and correct.

Background process subtraction



This mode enables the operator to monitor a process for any changes that may occur. Any difference in gas composition can easily be logged and displayed on the bottom "Difference" axis. The acquired spectra may be interrogated and compared with a spectra library for identification of new, evolved gas species at any time.

Full calibration Data Mode



This mode is the most commonly used mode. It enables the user to calibrate and display up to 64 gas species and measure in direct concentration. Each gas component is calibrated by a set up routine and once calibrated the instrument will measure, data log and display the data in real time. The above example shows a simple calibration and data logging of 4 common components that appear in air. All axis can be modified depending on the process and reaction you are wanting to measure.

Summery of Common Gas species V application

	N2	CO	CO2	O2	H2	H2S	CH4	C2H6	C3H8	C4H8	C5+
Bustle Gas	*		*		*		*	*	*		
Reformed Gas	*	*	*		*		*				
Process Gas	*	*	*	*	*	*	*	*			
Cooling Gas	*	*	*		*		*	*	*		
Feed Gas	*	*	*	*	*	*	*	*	*		
Reformer Flue Gas	*		*	*			*				
Natural Gas	*		*			*	*	*	*	*	*
Nitrogen Plant Gas				*							

The table above illustrates typical gas species for common process's within the Steel Industry. All above gas species may be measured using the Mass Spectrometer and data displayed in concentrations

Summery of Mass Spectrometer Analysis

- Fast flexible analysis technique
- Little specialised operator training required
- Low cost of ownership

Benefits to Steel industry manufacture and refinement:

- Optimise steel making process
- Maximise product production
- Minimise use of process gases such as Oxygen, Argon etc
- Safer operations
- Lower process emissions to atmosphere
- Greater consistency of product