Pure and Trace Gas analysis in the Bulk Gas, Fermentation, Food and Beverage Industries using Mass Spectrometry

Aspec. Application Note

Facts about aspec Mass Spectrometry

- ✓ Hundreds of gas's measured with one instrument
- ✓ Multi applications in one design
- ✓ Large dynamic ranges (9 decades)
- ✓ Fast analysis speeds. (ms).
- ✓ Very little Sample preparation.
- ✓ Compact, light weight robust design
- ✓ Low cost capital expense and cost of ownership
- ✓ PC based, Powerful, Comprehensive software
- ✓ Little specialised knowledge required

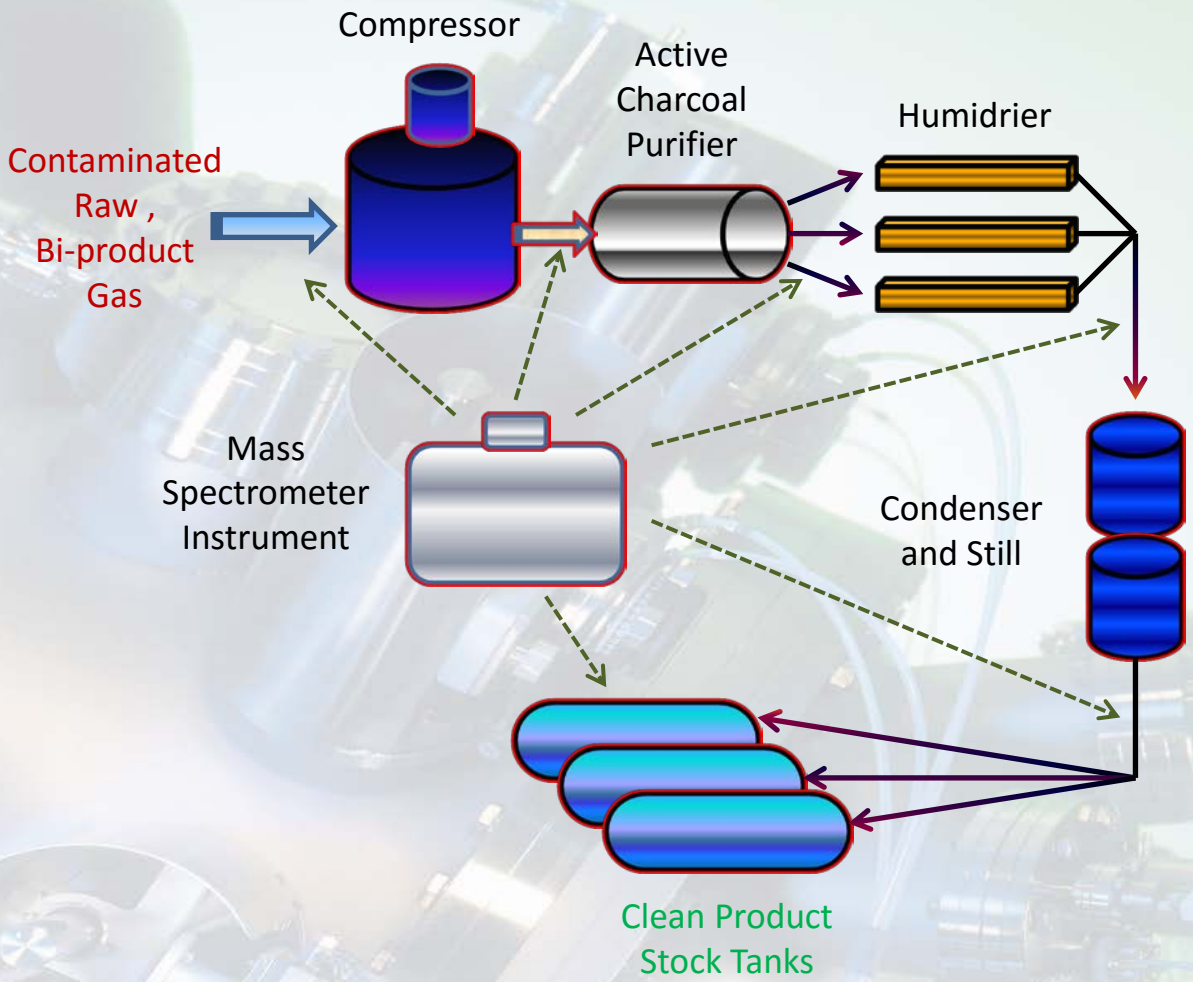
Laboratory and Industrial Scale

- Application areas:
- Bulk Gas Manufacture, Supply and Quality Control
- Chemical Processes and research
- Drinks Industry
- Fermentation Process and research
- Air Distillation process

- Example Application Areas
- Typical Gas/process Matrix
- MS analyser overview
- Software and System Architecture

Pure Gas Production process example

The Schematic shows a typical, complex Gas purification process used to supply pure CO2 and N2 gas into the worldwide food industry.
The Raw, potentially contaminated bi-product gas passes through a number of purification processes and eventually end up as clean gas.
The Mass Spectrometer may be used to continuously check all or any part of the process not only to check on the products improving quality but to check the performance of each part of the process itself.
Some of the process parts like the Active Charcoal Filter may require “re-generation”. The Mass Spectrometer will allow the operator to determine if and when this is required often optimising process up-time and reducing operating costs.
Often high levels of Water are often present during the purification process this poses no a problem for the Mass Spectrometer instrument!



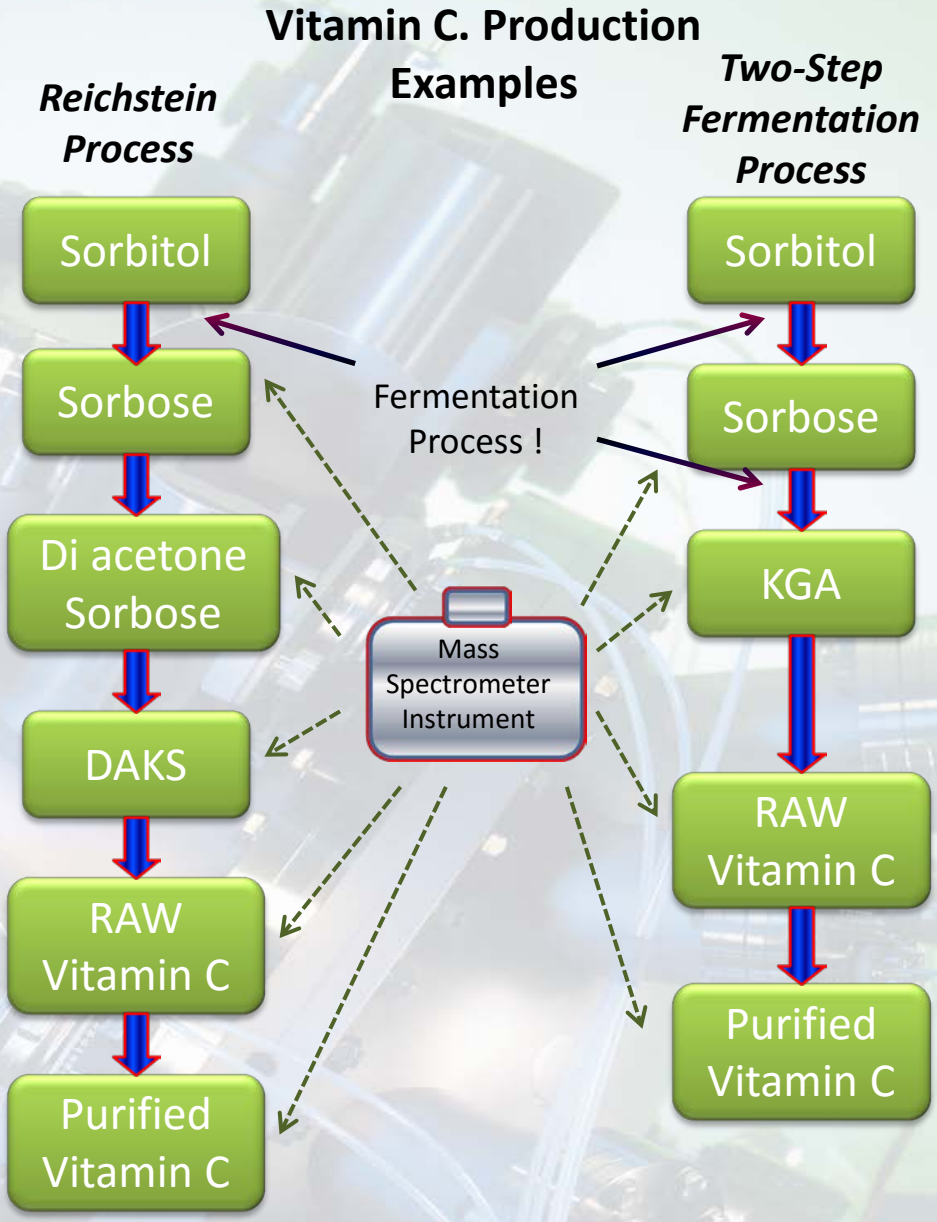
Typical use of MS in Fermentation:

Fermentation Processes where MS may be used:

- Fermentation Food/Wine
- Fermentation Biochemistry
- Ethanol Fermentation
- Industrial Fermentation
- Lactic Acid Fermentation
- Fermentation Hydrogen Production
- Sewage Disposal Fermentation.

Vitamin C example:

The MS may be used to monitor and measure many points in the production processes and also “trouble shoot” process activities.
The MS has a fast response and ultra low detection limit and may be utilised to measure gas components used in the Vitamin C production process such as:
Acetone, Chlorine, Sodium Hydroxide, Sulphuric Acid, Acetic Acid, Butyric Acid, n-Butanol , Ethanol.
Methane may also be measured routinely using MS as this often indicates contamination and may be treated with Antibiotics to preserve the process batch.



Principles of the Mass Spectrometer

Sample gas is continuously injected into a the vacuum housing held at approximately 1 x 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.

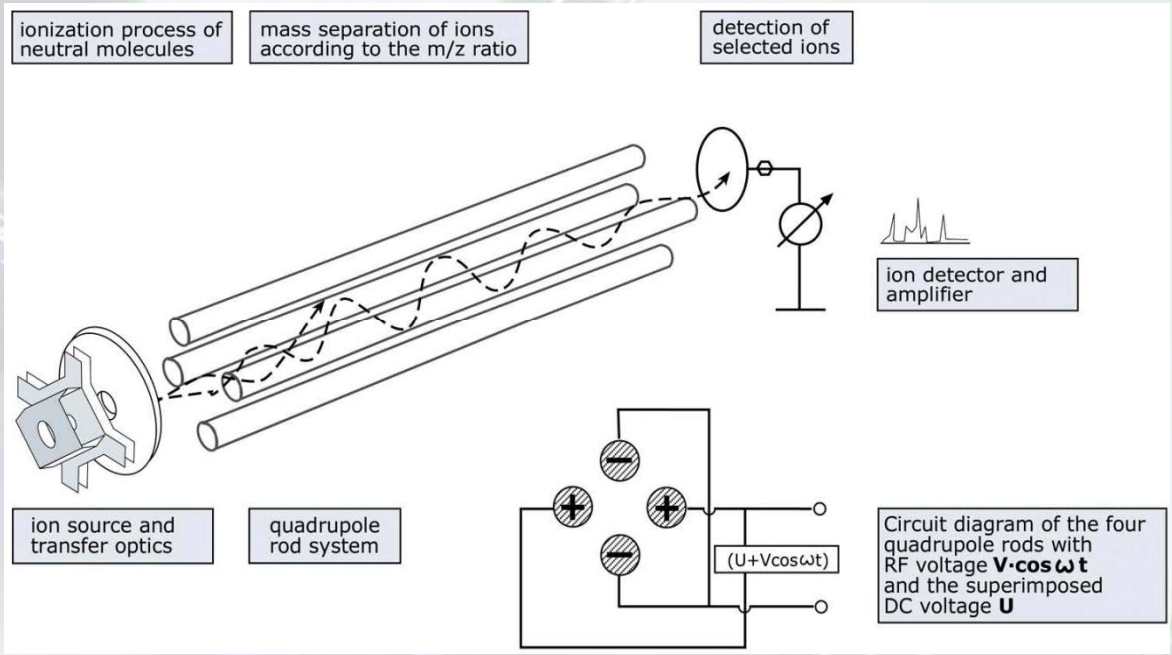
These ions are focused from the Ion Source via transfer optics towards the mass filter (quadrupole rod system). The Rod System consists of 4 accurately ground stainless Steel rods mounted very accurately into a set of ceramic carries.

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.



Mass Spectrometer Analysis

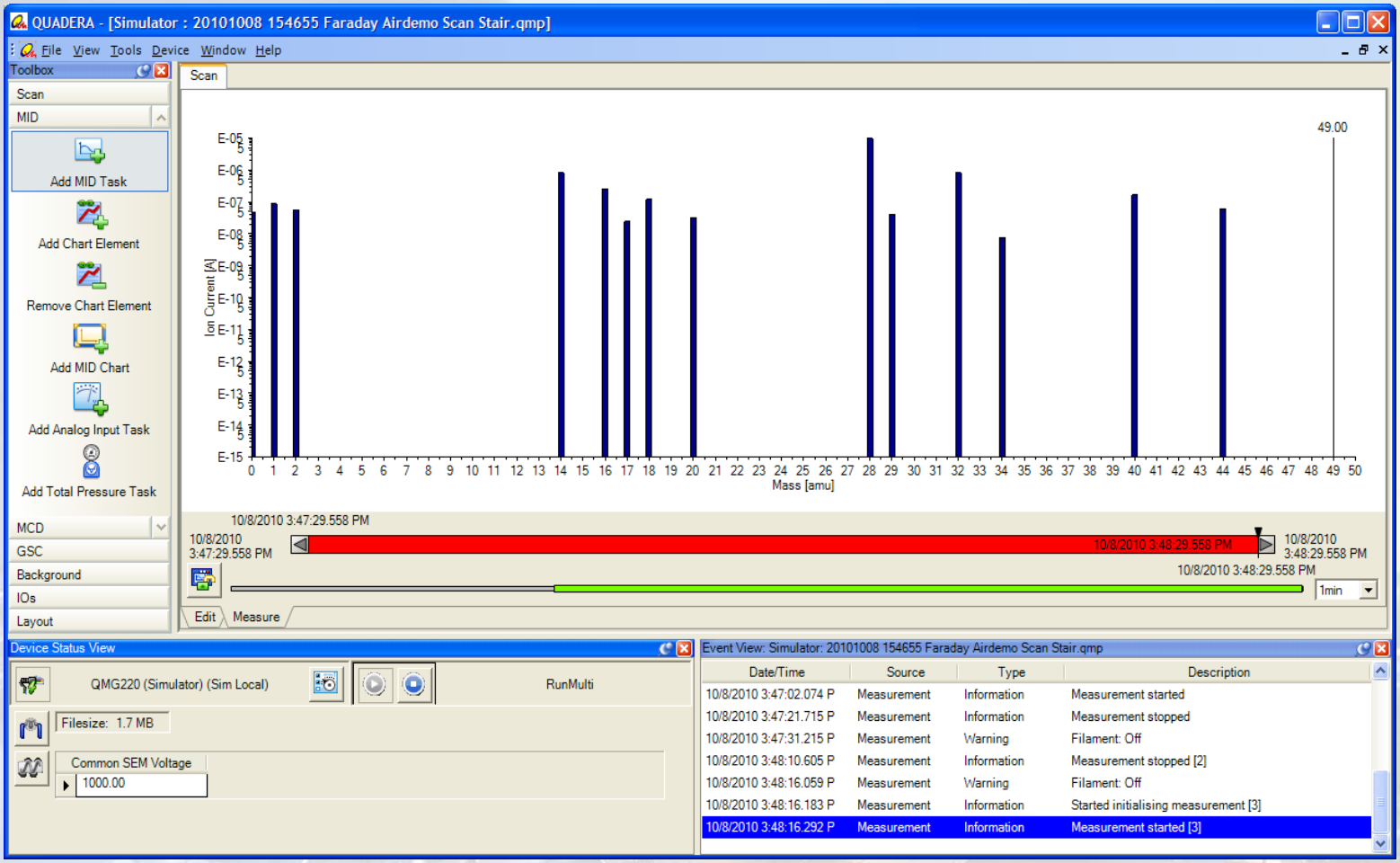
The Mass Spectrometer instrument is an extremely fast and flexible instrument and has the capability to measure up to 64 gas species at any one time.

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

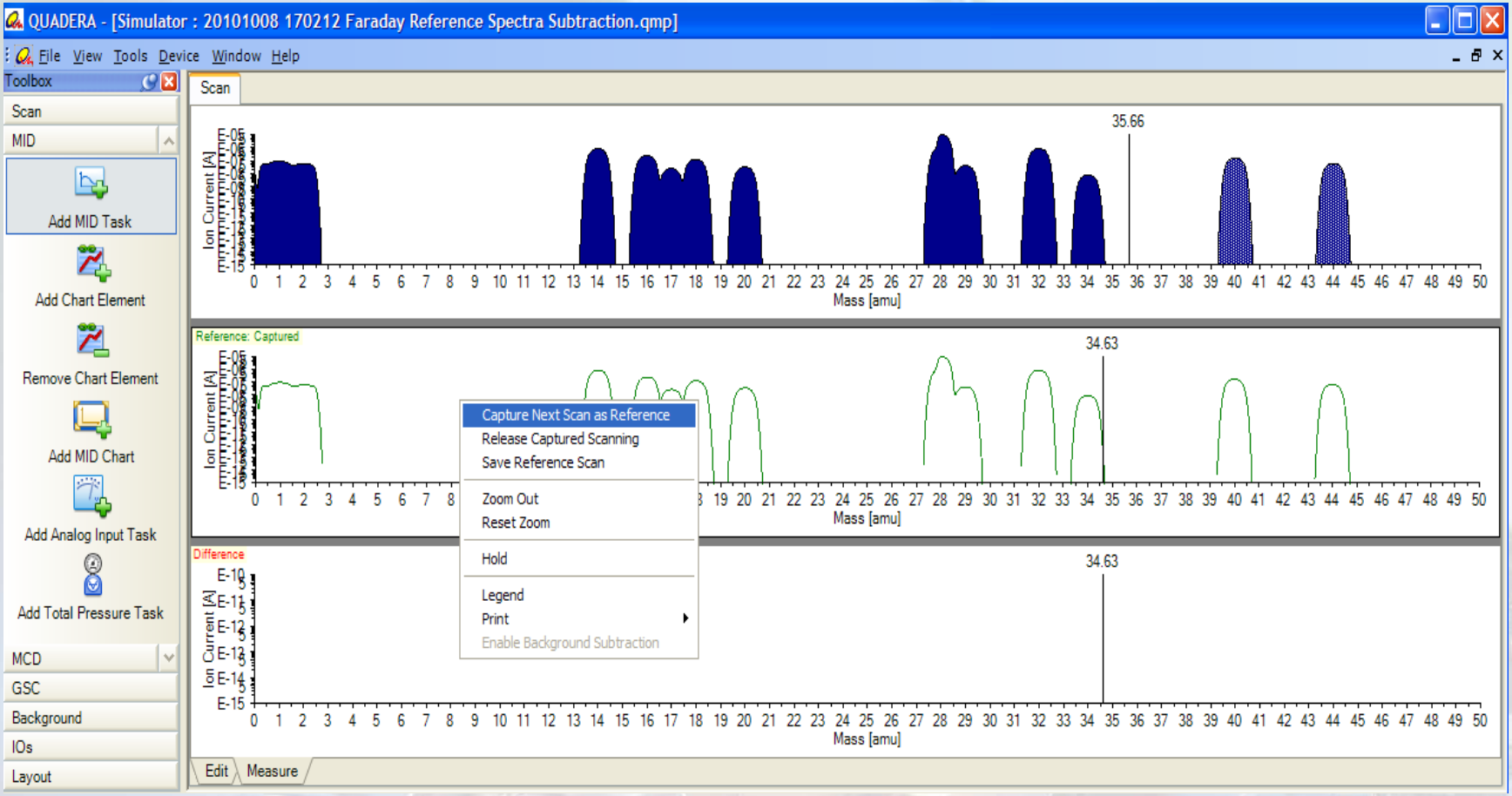
Raw Gas/Mass scan Mode



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.

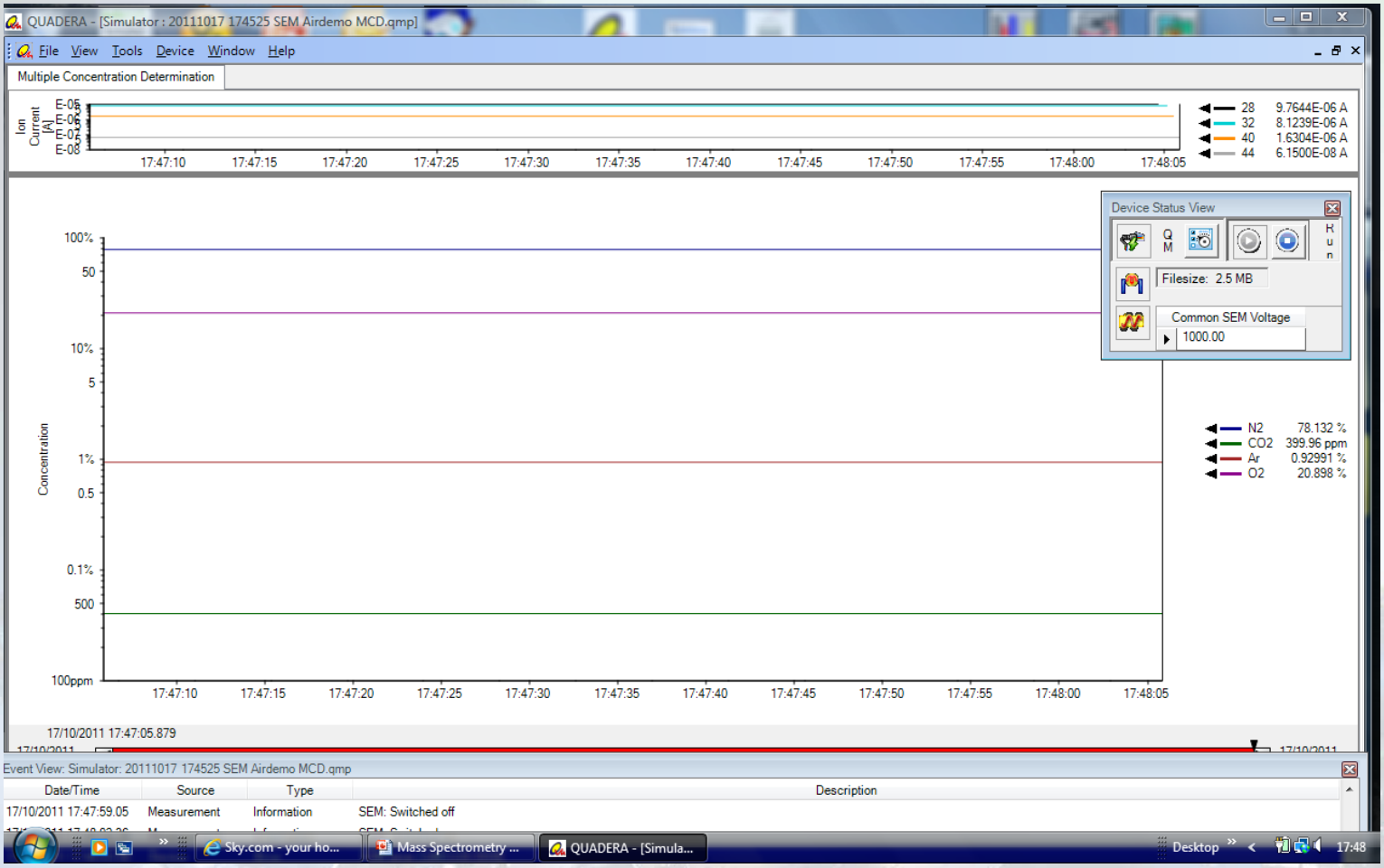
This mode enables the user to quickly see if the gas composition is accurate and correct.

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 the 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.

Typical Common Gas species in the Pure Gas Industry:

Gas Type	Range
Argon	10ppm to 100%
Acetylene	0.5ppm to 100%
CO2	1ppm to 100%
Helium	1ppm to 100%
Hydrocarbons	10ppb to 100%
Hydrogen	50ppm to 100%
Nitrogen	1ppm to 100%
Oxygen	0.5ppm to 100%

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

Typical Common Gas species in the Fermentation Industry:

Gas Type	Range
Ethanol	0.5ppm to 100%
CO2	5ppm to 100%
Nitrogen	1ppm to 100%
Oxygen	10ppm to 100%
Water	100ppm to 98%
Methane	10ppm to 50%
Hydrocarbons	ppm to 100%
Xylene	10ppb to 100%
Toluene	10ppb to 100%
Styrene	10ppb to 100%
Acetone	50ppb to 100%
Chlorine	ppm to 100%
Sodium Hydroxide	ppm to 100%
Acetic, Butyric, Sulphuric Acid	ppm to 100%

Typical Common Gas species in the Food and Beverage Industry:

Gas Type	Range
SO2	10ppb to 100%
MIBK	10ppb to 100%
Methanol	10ppb to 100%
MEK	10ppb to 100%
H2S	10ppb to 98%
Ethanol	10ppb to 100%
DMS	10ppb to 100%
DME	10ppb to 100%
COS	10ppb to 100%
Benzene	5ppb to 100%
1-3 Butadiene	10ppb to 100%
CO2	1ppm to 100%

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

Summery of Mass Spectrometer Analysis within the Pure Gas, Fermentation and food and Beverage Industries

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

Benefits to Pure Gas industry manufacture, supply, research and refinement:

- Very Fast Continuous measurements of many potential contaminants
- Continuous measurement of product quality
- Ability to scan for unknowns
- Ideal for Process investigation and optimisation
- Multi application instrument