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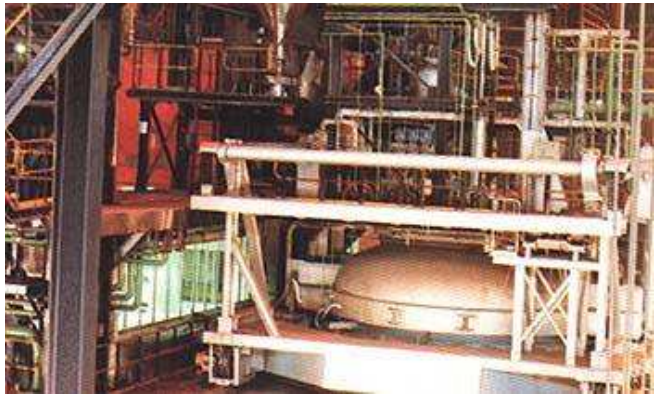
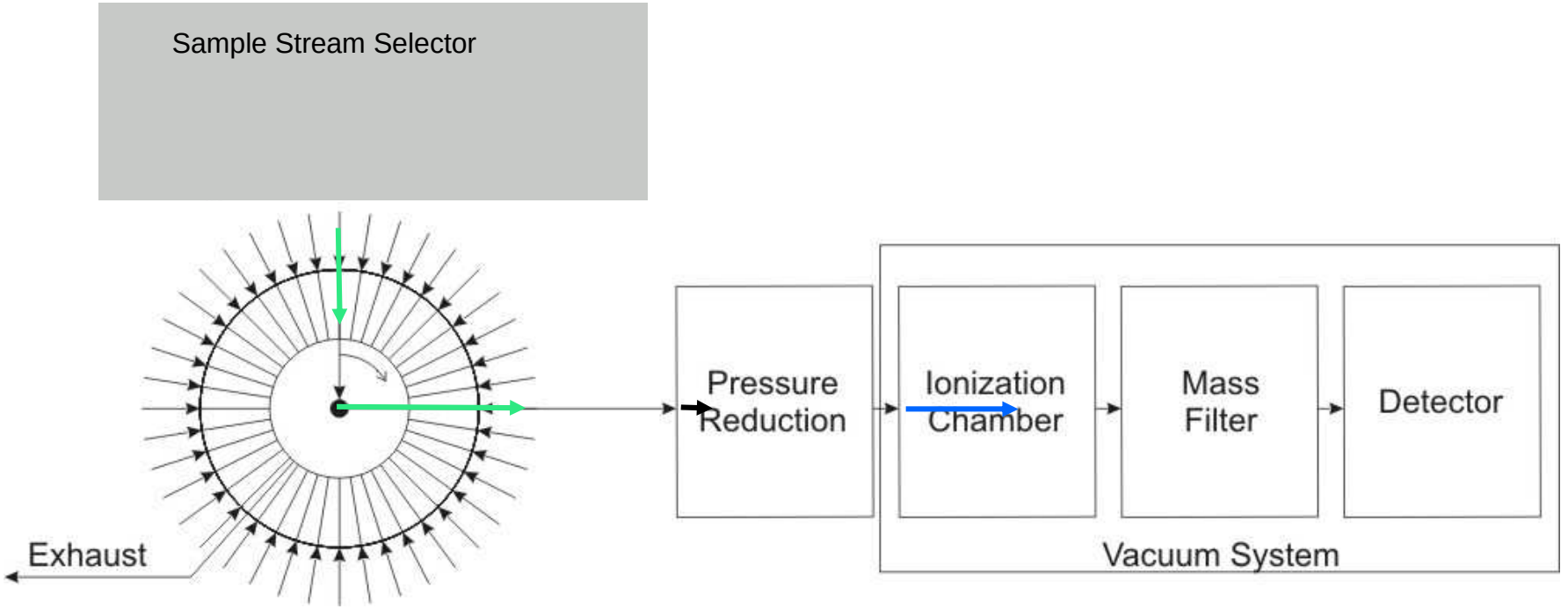
Applications of a Magnetic Sector Process Mass Spectrometer to the Analysis of Variable Vacuum Samples



Outline of Presentation

- Introduction
  - Process Gas Analysis Magnetic Sector Mass Spectrometer
  - Standard types of Inlet
  - Performance
  - Effects of Variable Pressure
- VOD/RH Gas Analysis
  - Special Inlet
  - Results
- Multi-Stream Vacuum Drying Gas Analysis
  - Special Inlet
  - Results

Gas Analysis Mass Spectrometer

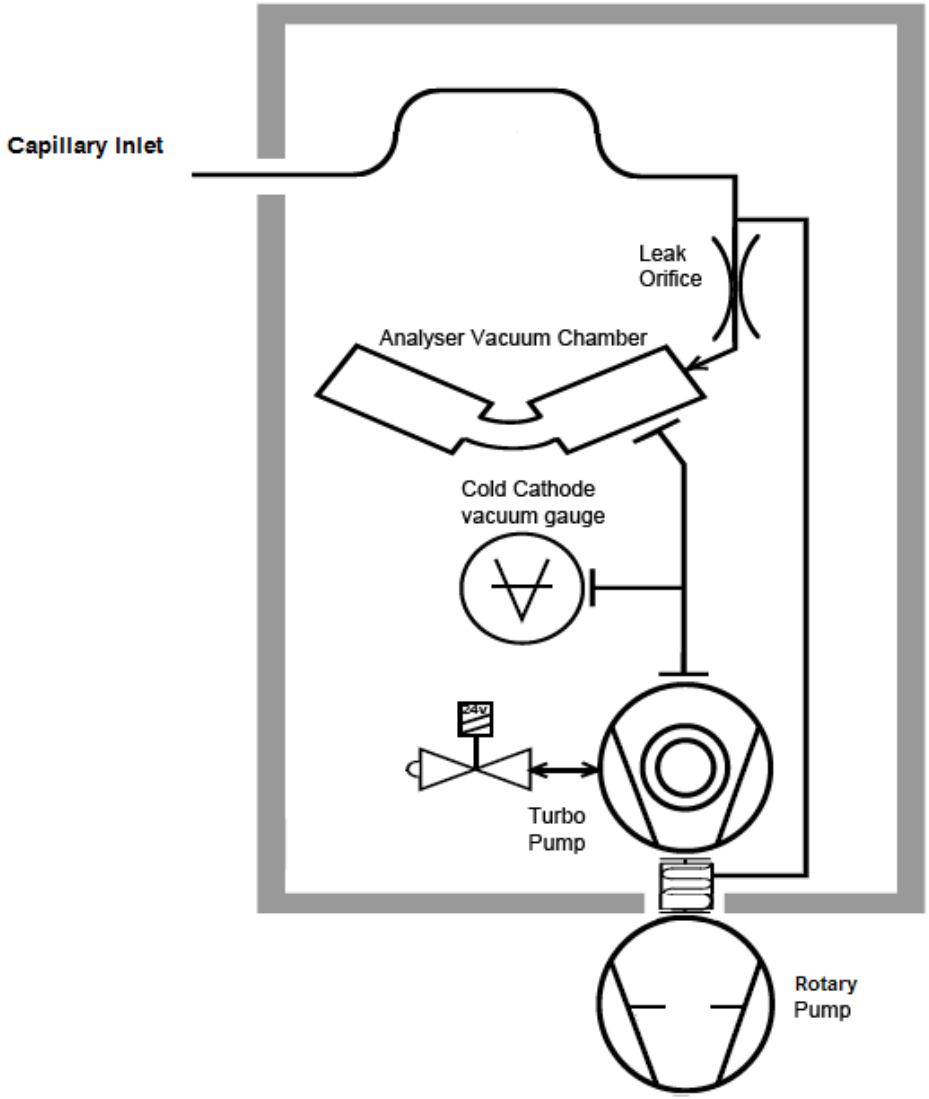


Typically sample gas at slightly above atmospheric pressure

Inlet reduces pressure to ~10-4 mbar

Ions are separated according to mass by electric and/or magnetic fields

Capillary Inlet



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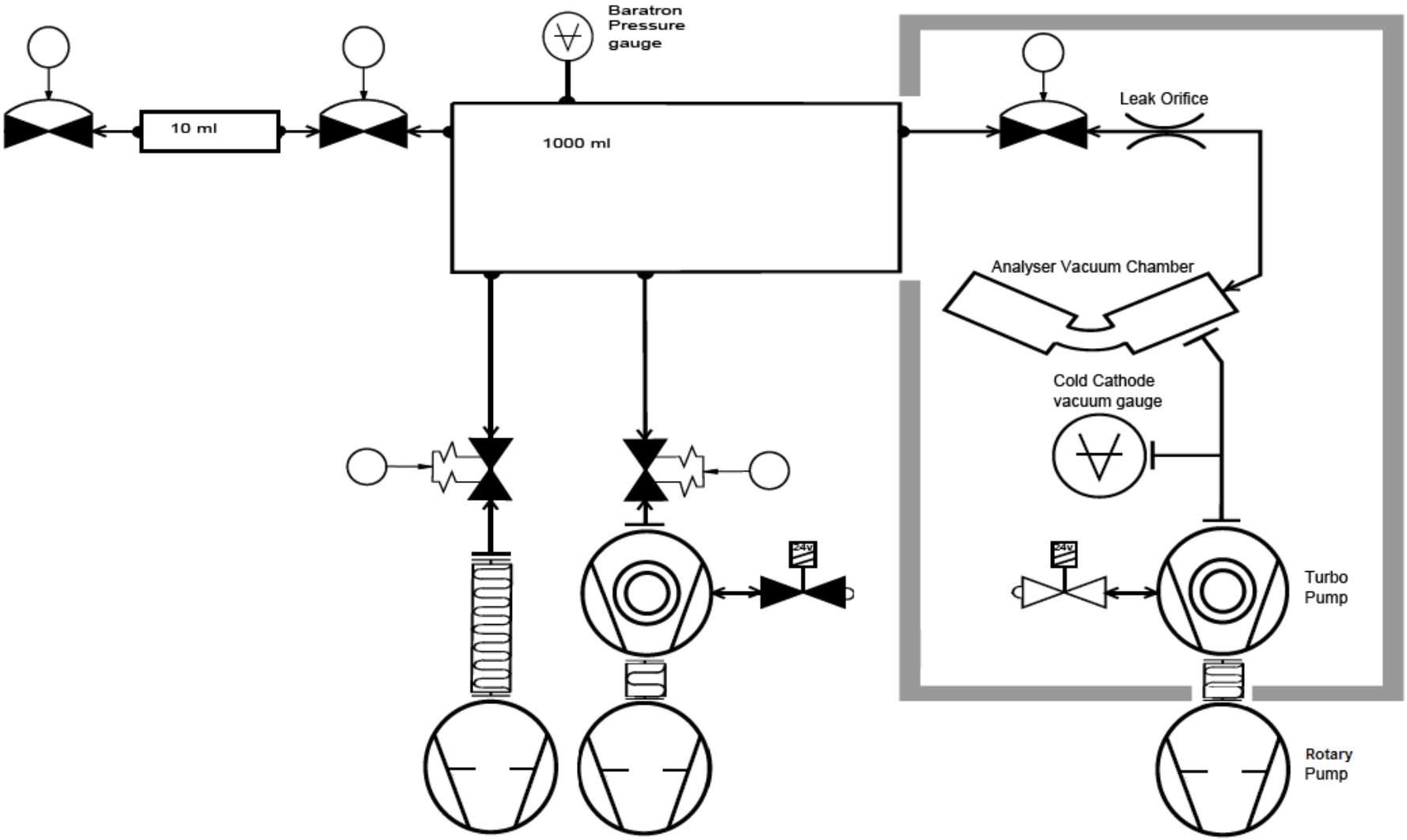
Batch Inlet



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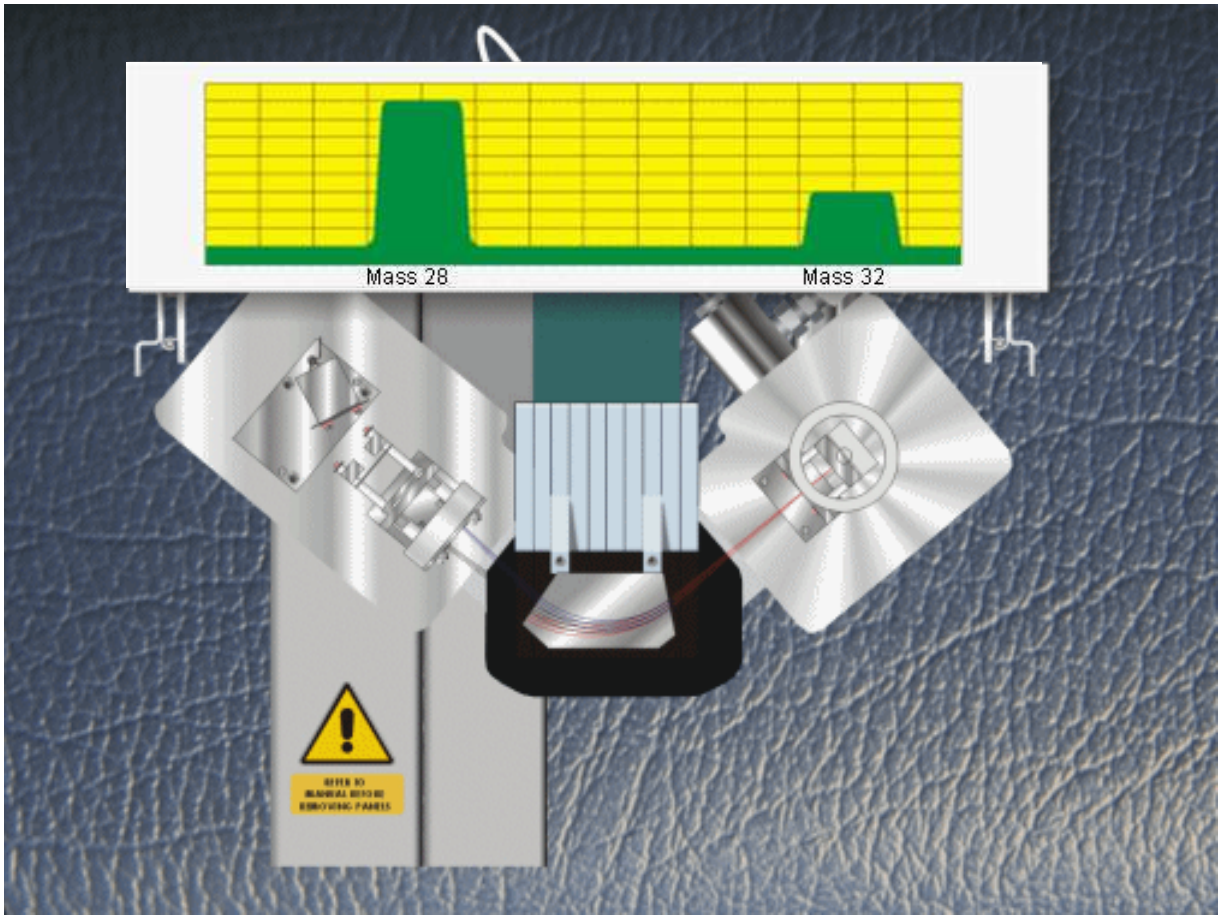
Scanning Magnetic Sector – principles of operation



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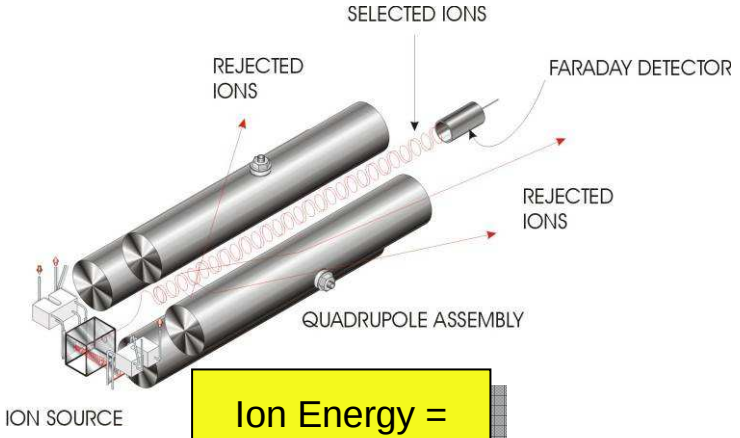
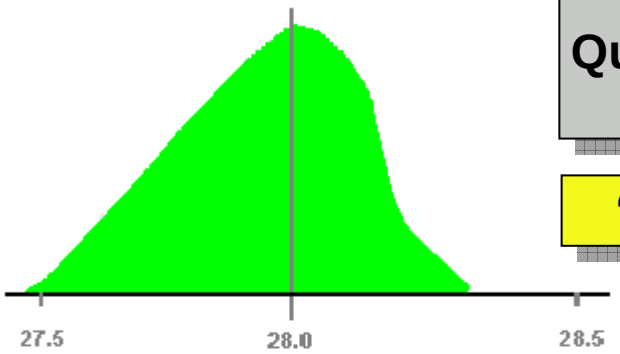
Quadrupole / Magnetic Sector – contrast

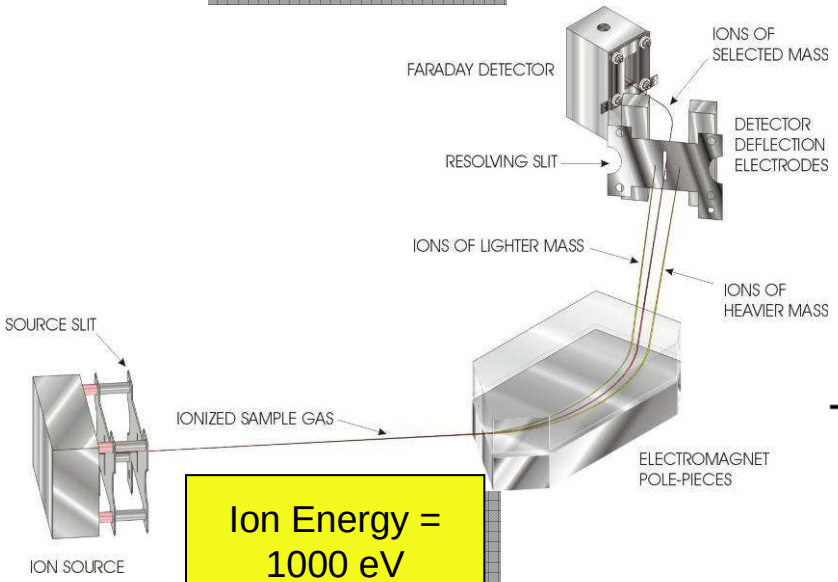


Ion Energy = 5-10 eV

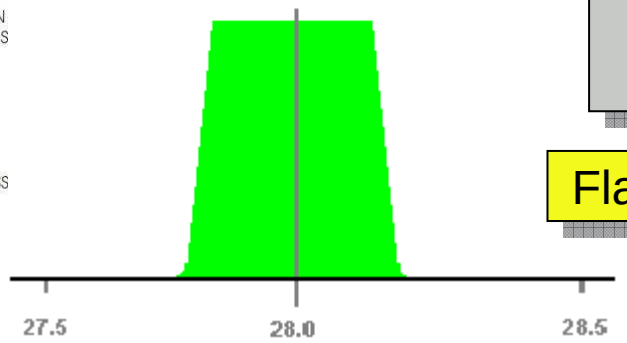


Quadrupole MS

'Pointed' Peaks



Ion Energy = 1000 eV



Magnetic MS

Flat-topped Peaks

Ultra-High Stability

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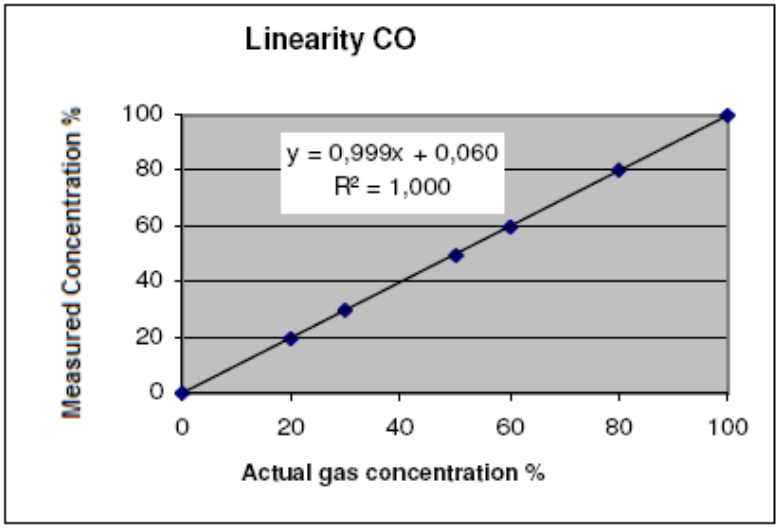
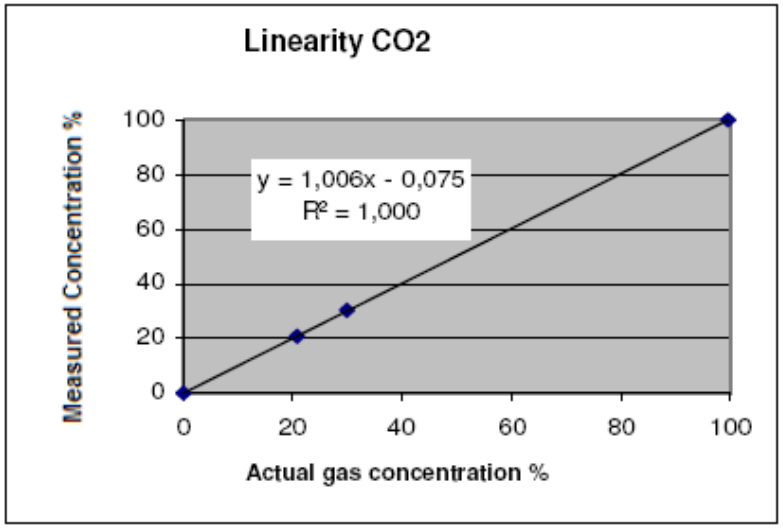
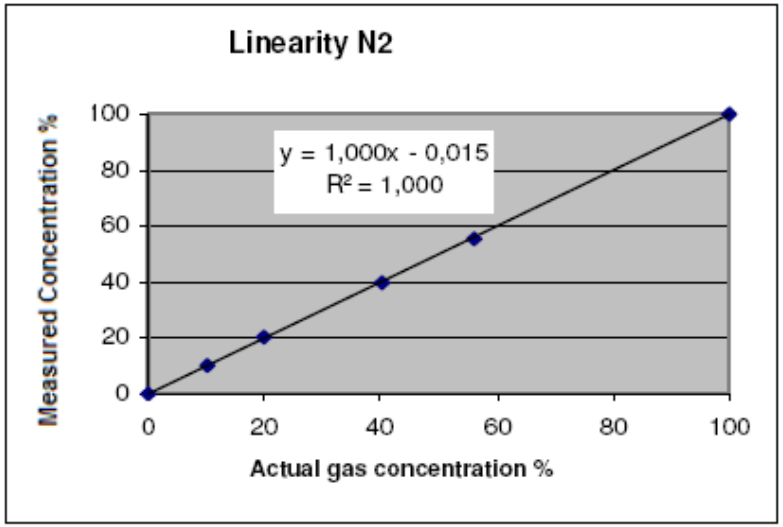
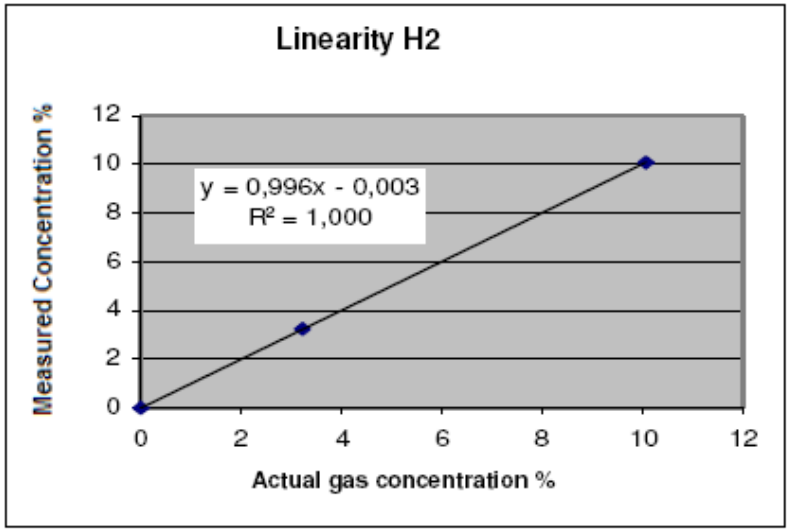
Analytical Accuracy - Linearity









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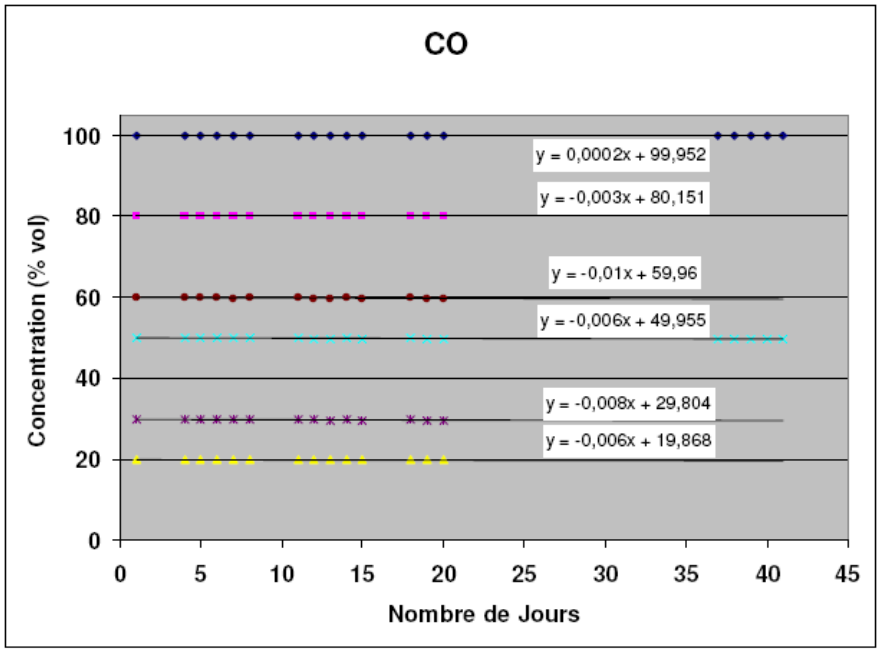
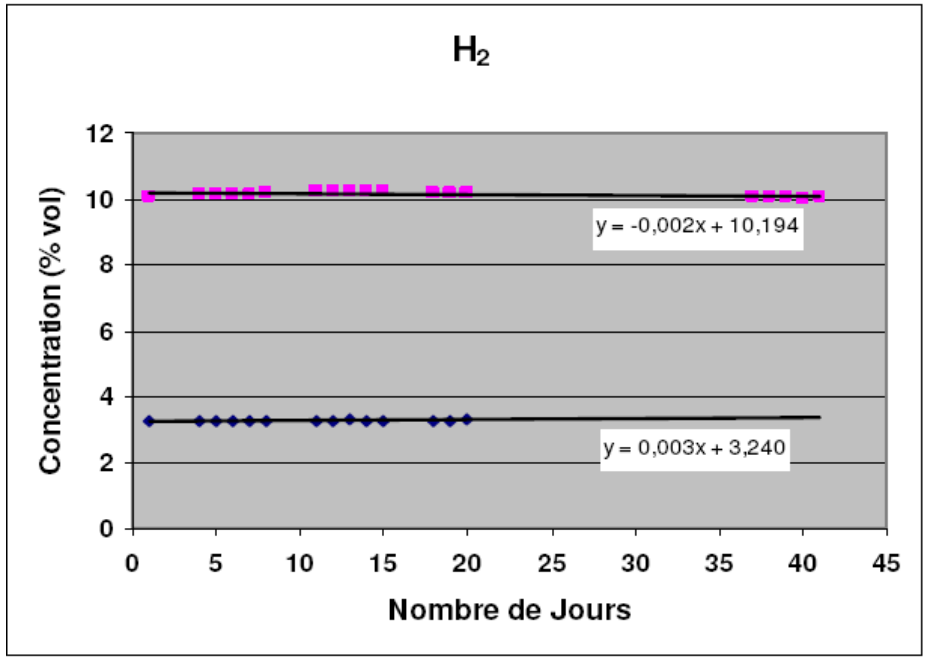
Analytical Accuracy - Stability





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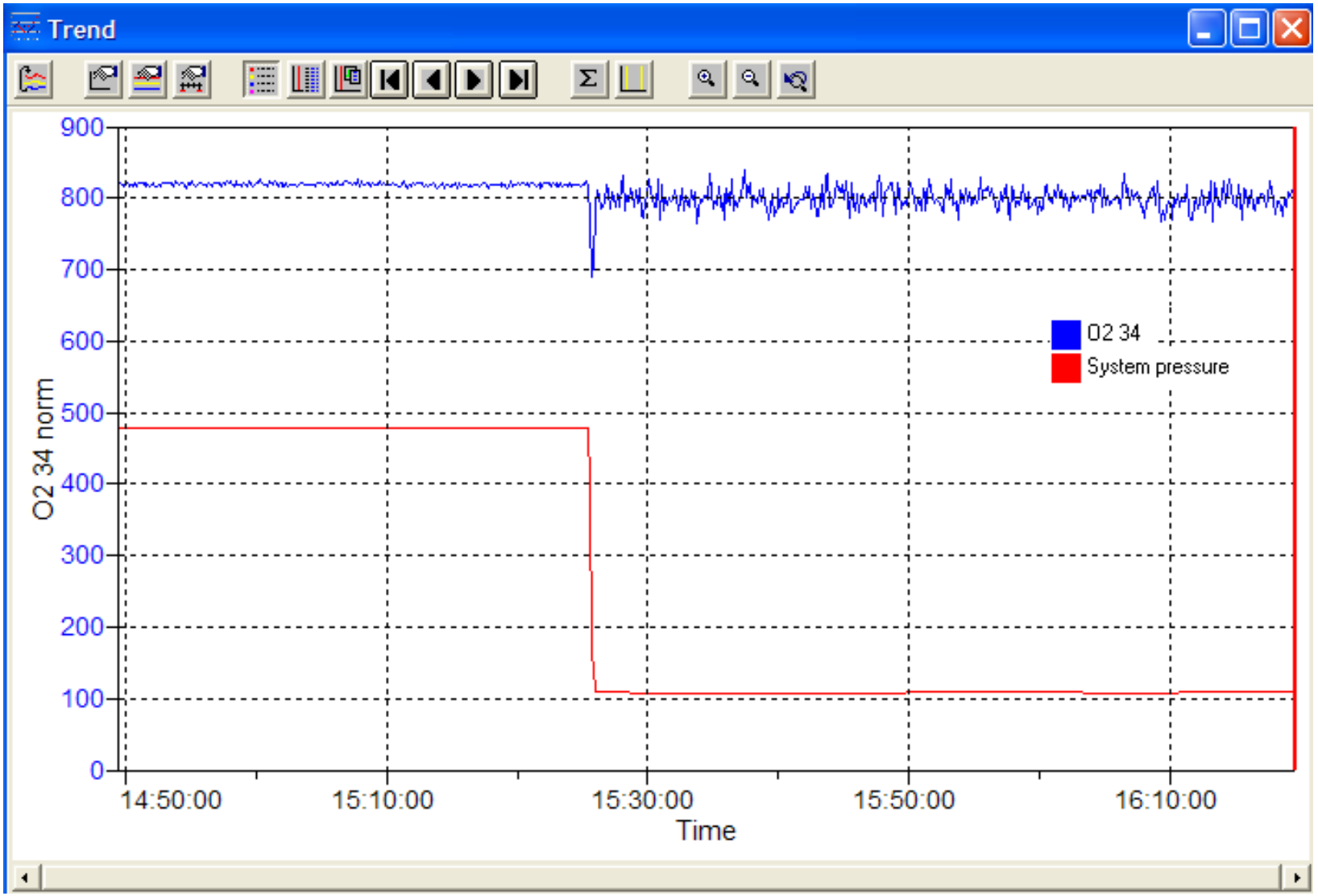
Possible Problems with Variable Pressure Samples

- Compromised Dynamic Range
- Non-linear Analysis

Effect of Pressure on Repeatability



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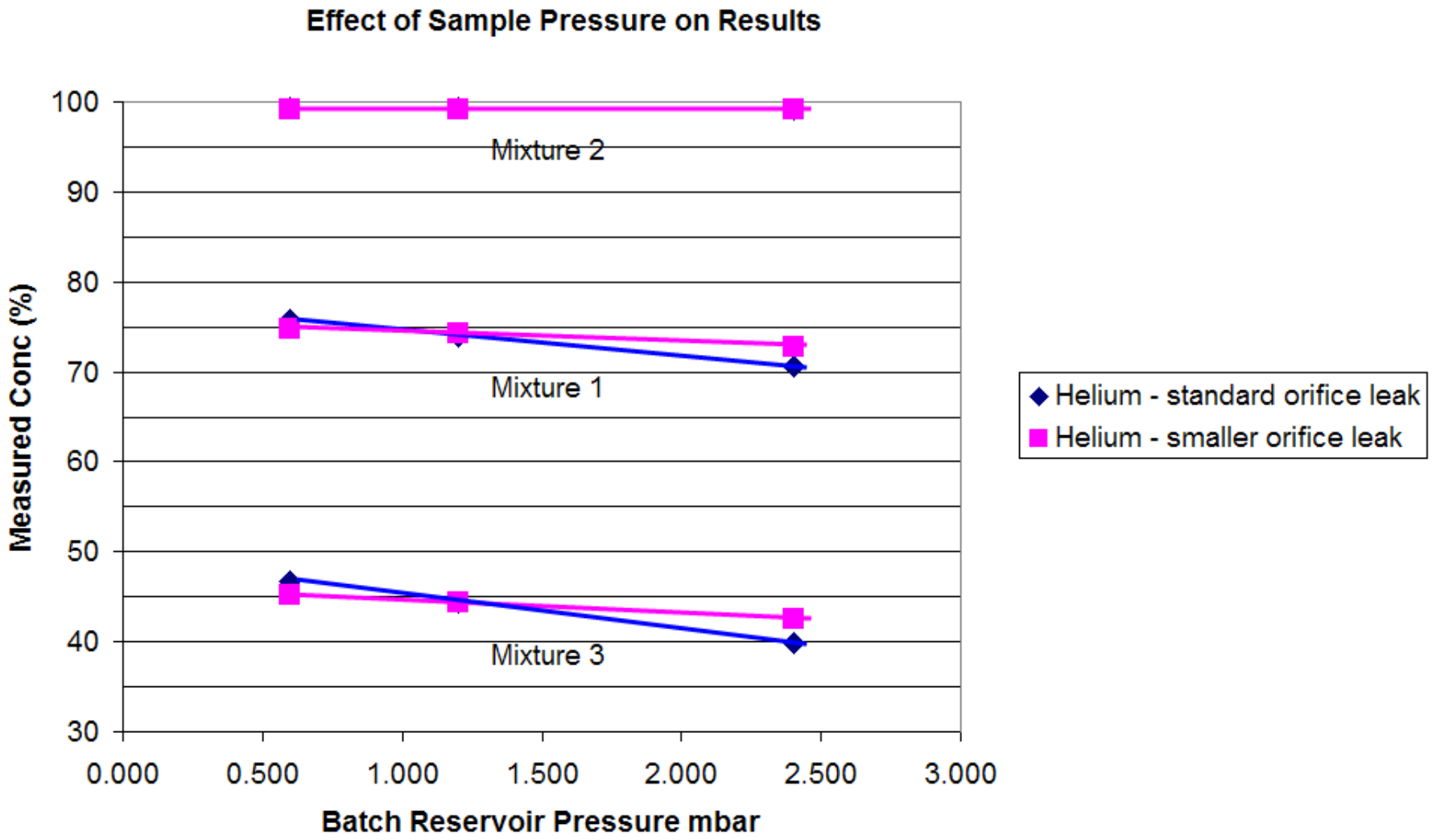
Non-Linear Effect of Pressure



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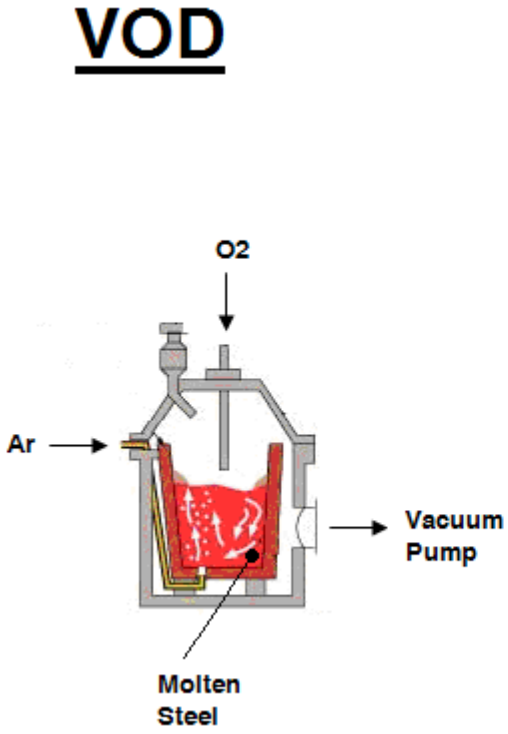
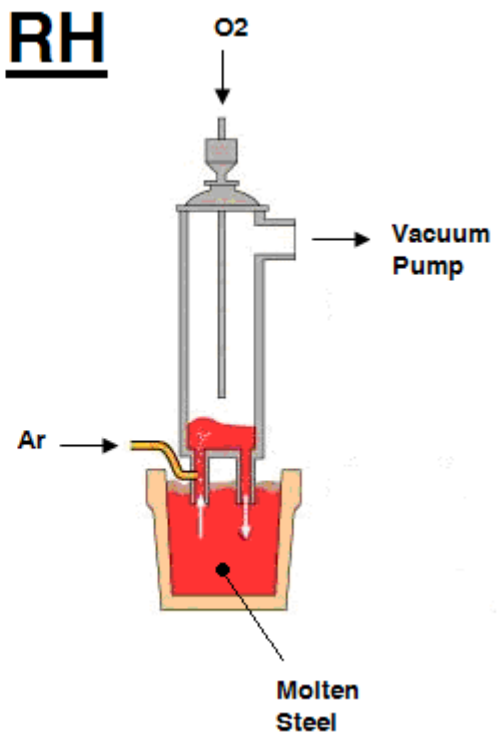
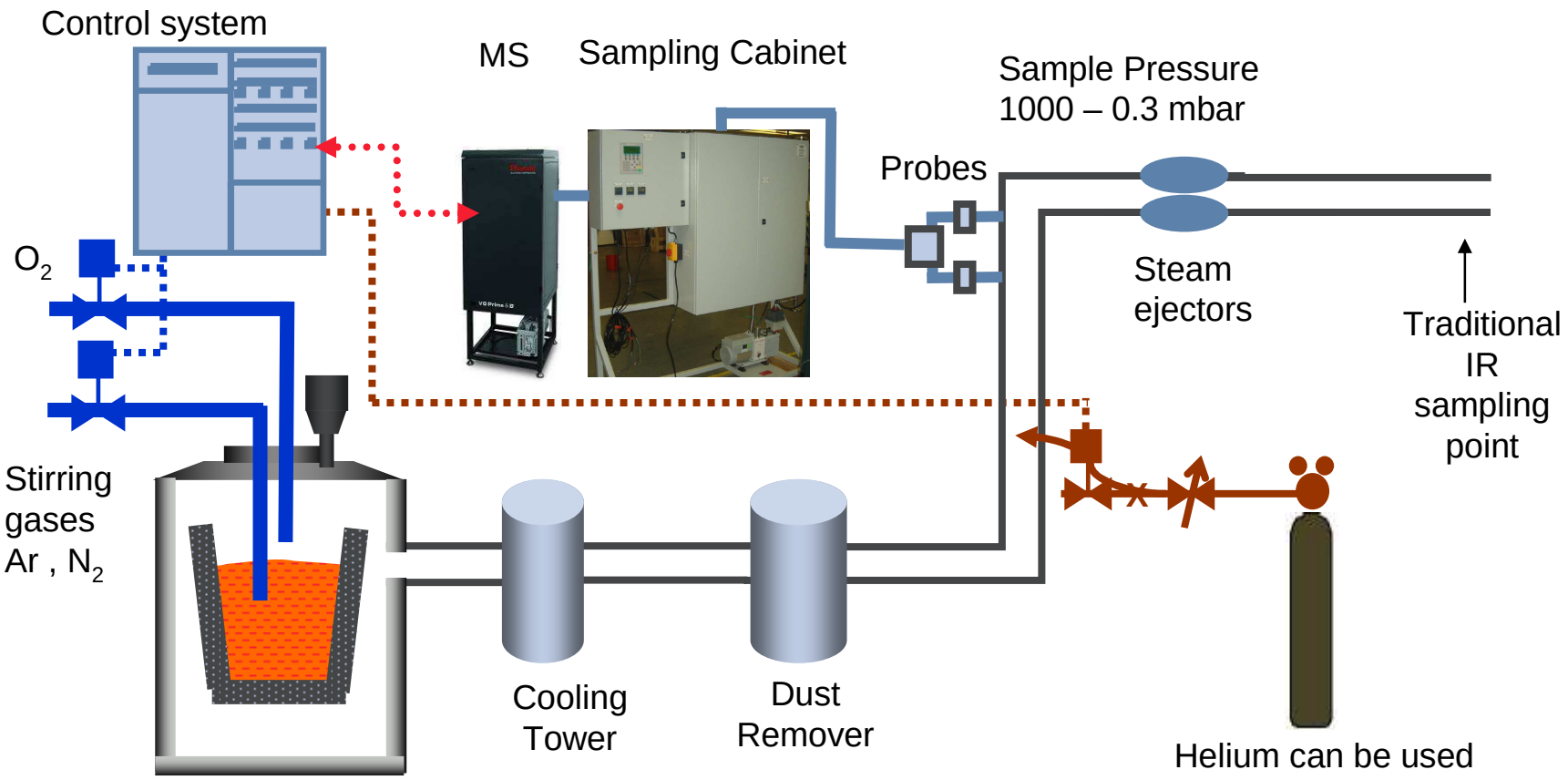
VOD and RH Vacuum Degassing Processes





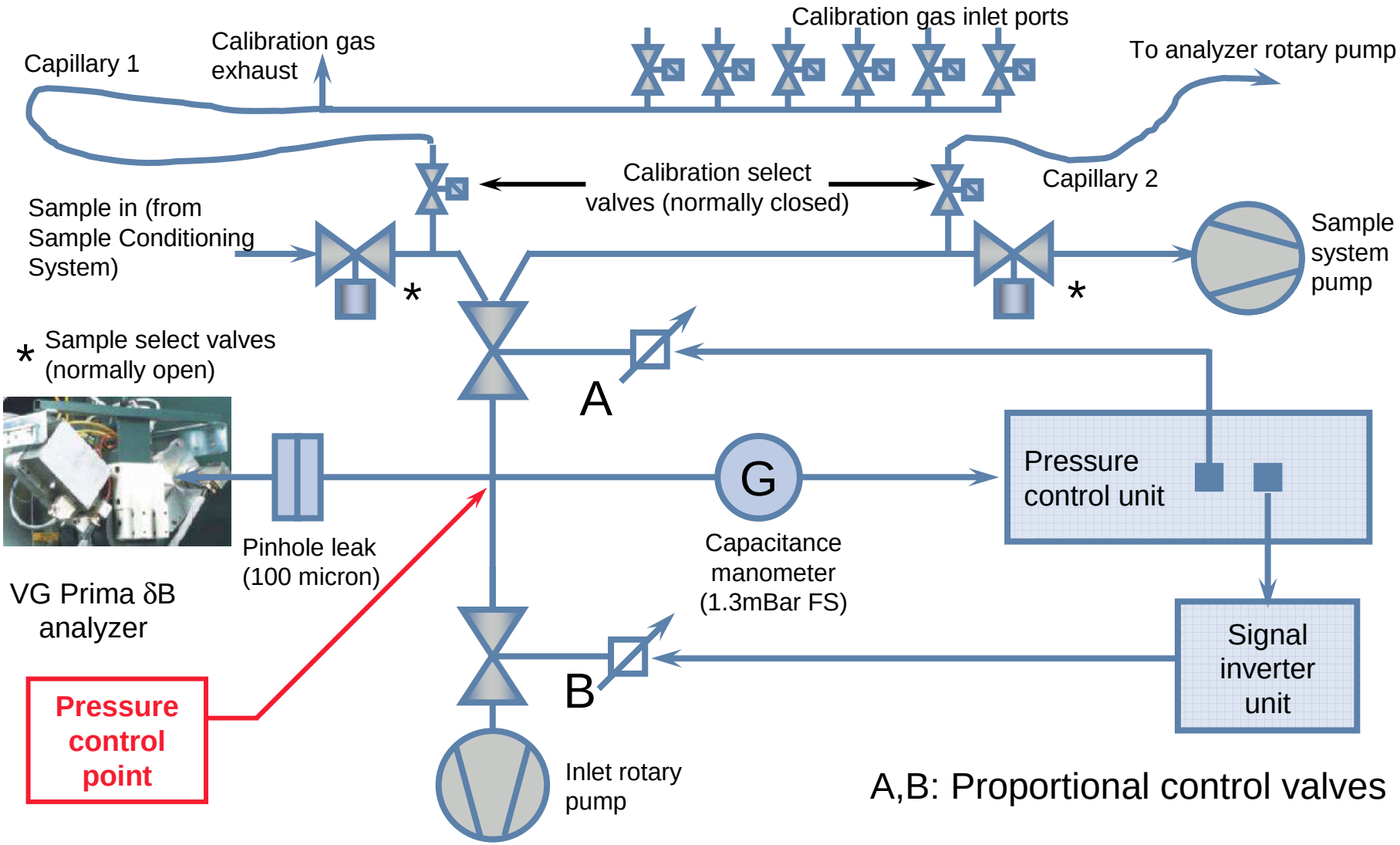
Processes used to make speciality steels, such as stainless steels

VOD or RH Process Gas Analysis with Mass Spectrometer



MS measures CO, CO_2, O_2, N_2, H_2 and Ar process off-gases, and He tracer gas

Magnetic Sector Prima δB VOD/RH Inlet



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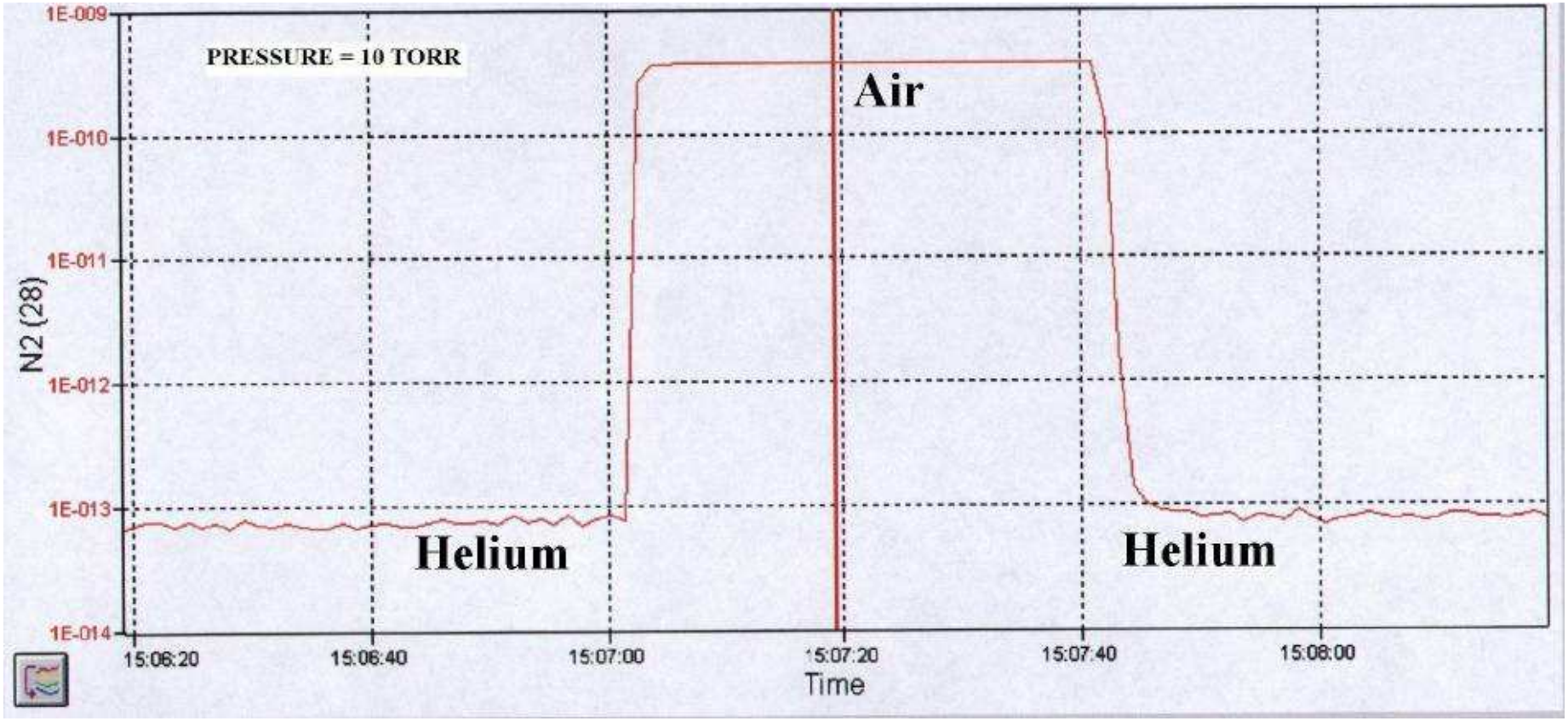
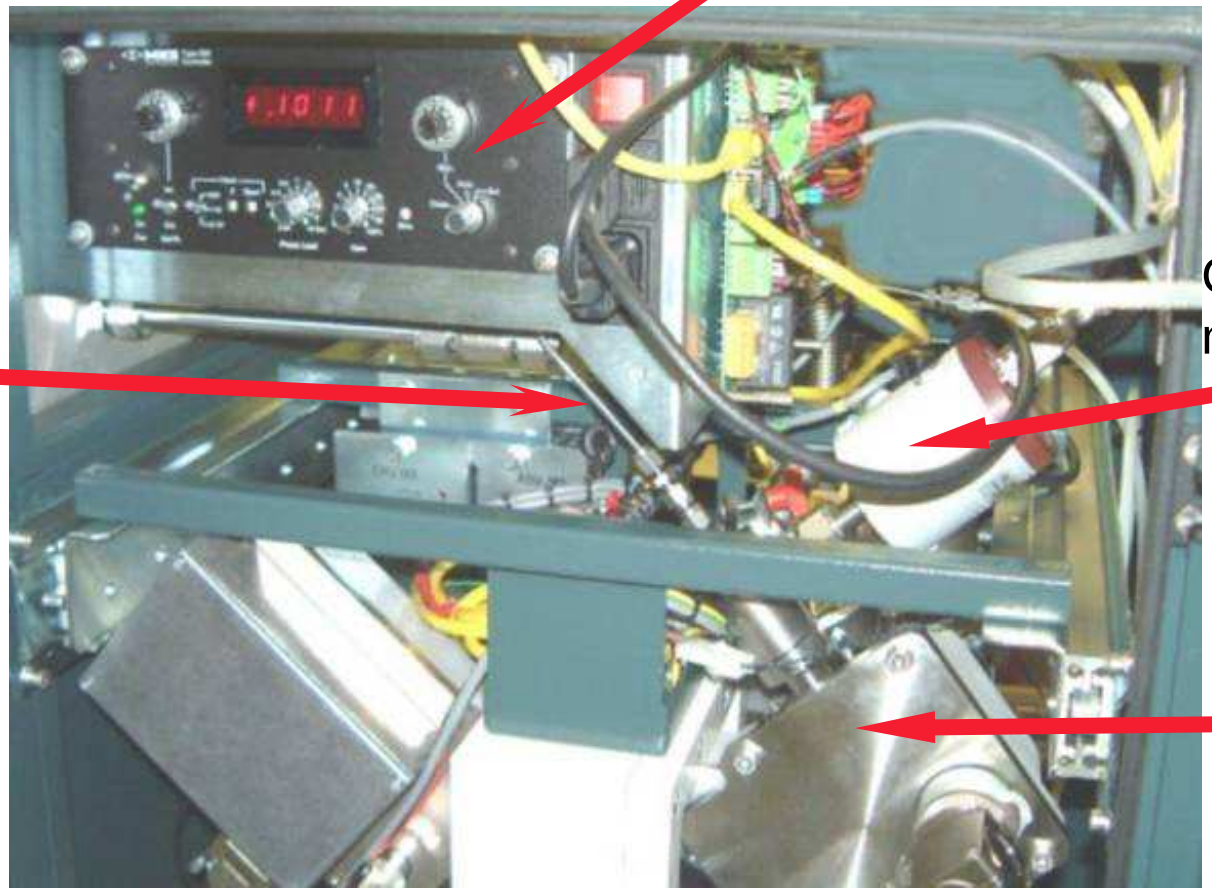
Demonstration of Response Time of SCS + MS



This is achieved by high pumping speed using a large capacity vacuum pump and low internal volume of pipe-work between sample probe and MS analyzer

Prima δB VOD/RH Inlet



Prima δB VOD/RH Performance Specification

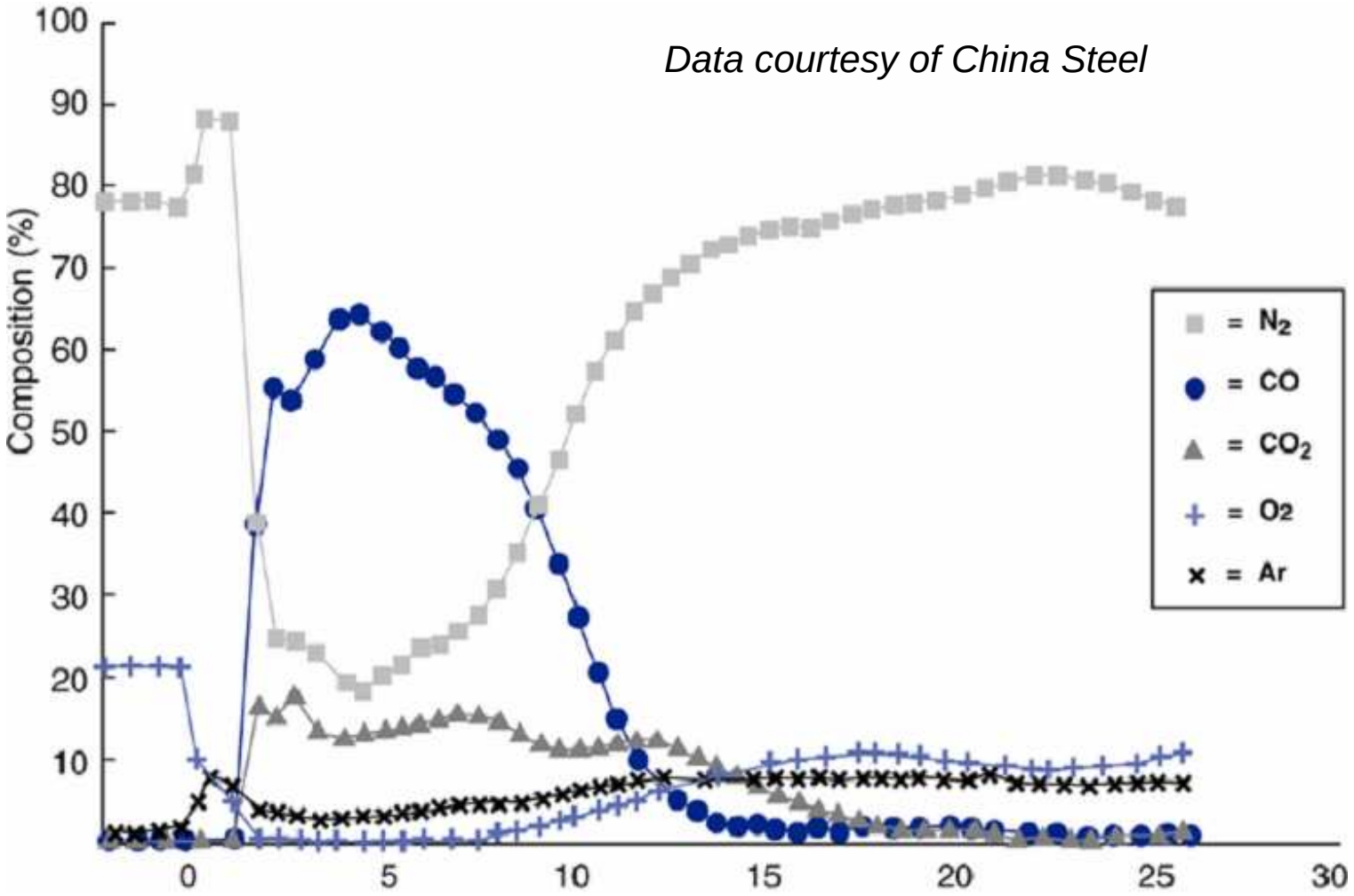
	% Molar concentration	Precision % absolute
H_2	2	0.005
He*	1	0.005
CO	25	0.03
N_2	53	0.05
O_2	2	0.002
Ar	2	0.002
CO_2	15	0.02

- Precision is standard deviation over 8 hours
- Analysis time: 6 seconds
  - Analysis can be reduced to 3 seconds, precision reduced by factor of 2

** Helium measured if used as tracer gas*

RH Process Data

Data courtesy of China Steel



Vacuum pressure ⟶ 5 torr 1 torr 0.5 torr

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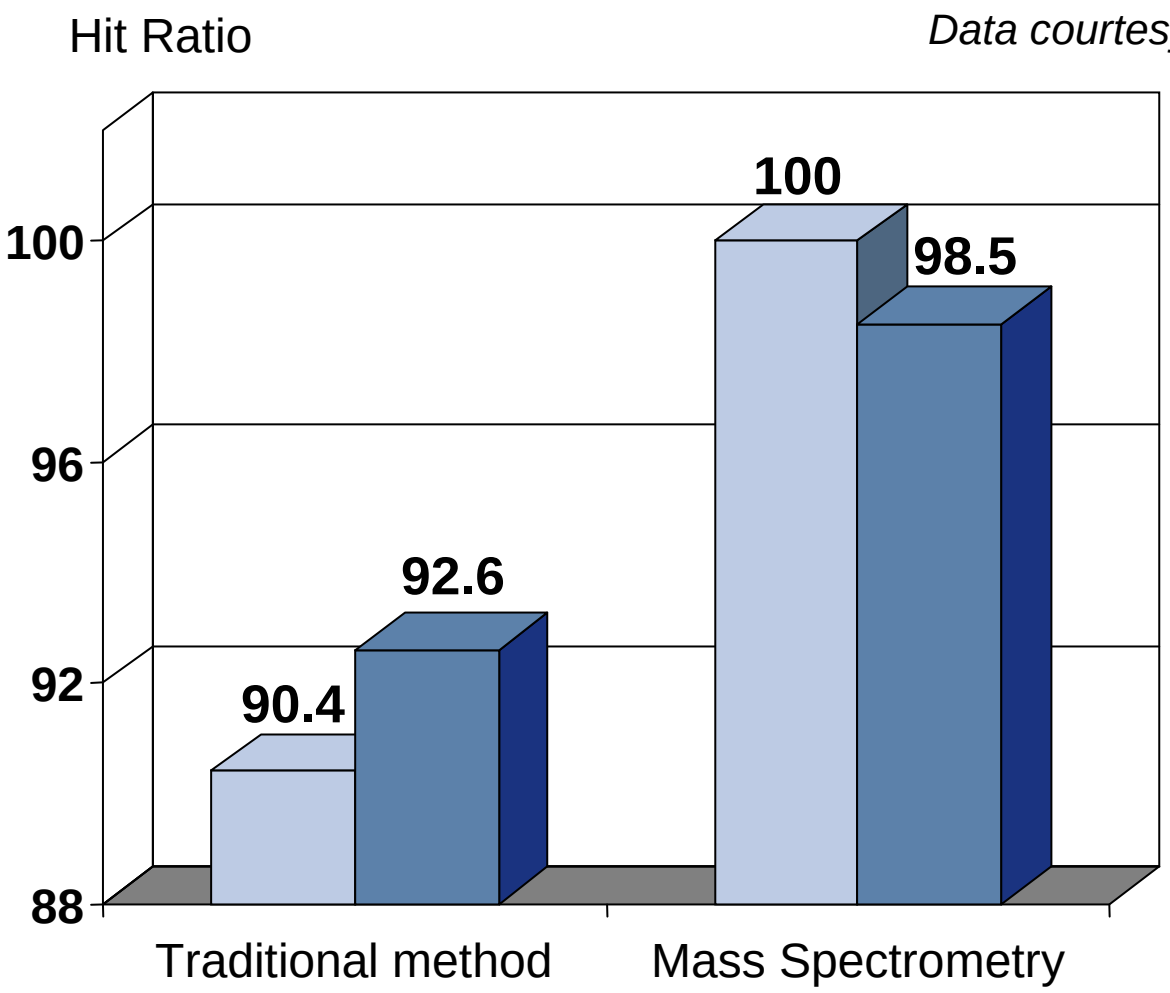
RH Process Hit Ratio

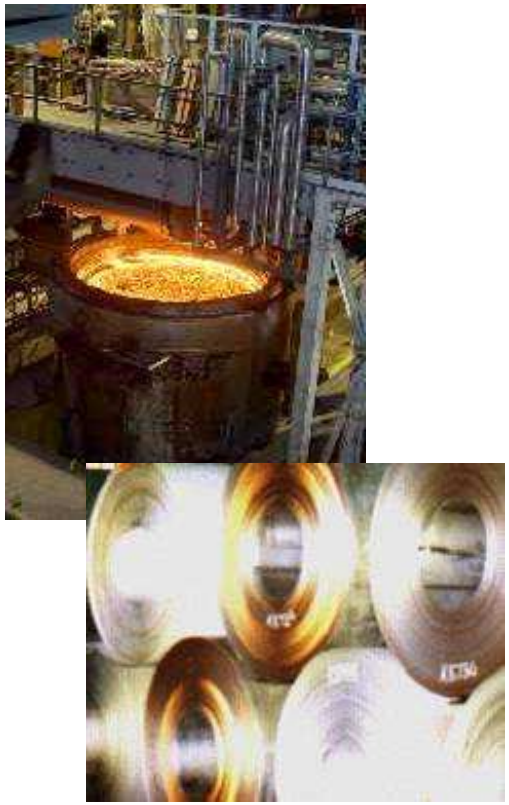


Data courtesy of China Steel

30 ppm steel
150 ppm steel

- With Thermo MS
  - *For ≤ 30ppm C steel, mean error ~1.9ppm*
  - *For 150~250ppm C steel, error ~ 13.6ppm*

Summary



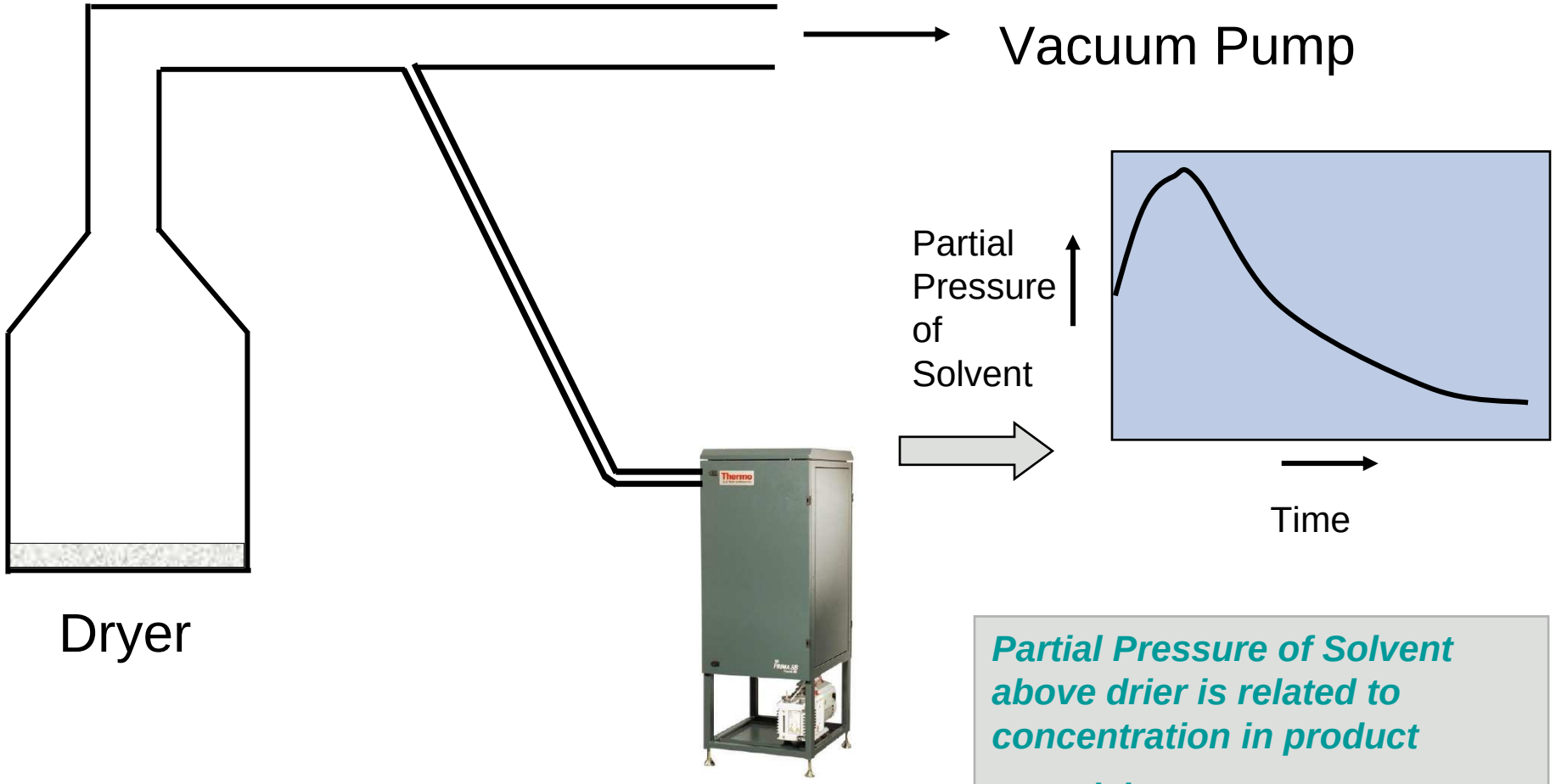
- Prima δB has been used with great success for RH and VOD processes
- Offers fast, precise complete off-gas analysis
- Unique variable pressure inlet enables system to monitor RH and VOD processes over wide range of pressures (~1000 mbar – 0.3mbar)
- Enables faster production of higher quality steel

Monitoring of Solvent Drying Process

- Traditionally performed by off-line analysis – involves interrupting process and performing laboratory analysis of extracted sample (by e.g. HPLC, GC, NMR or TG)
  - Time consuming and labour intensive and may not be representative
- Risk of over-drying
- The above wastes Time & Energy and can possibly reduce Product Quality!

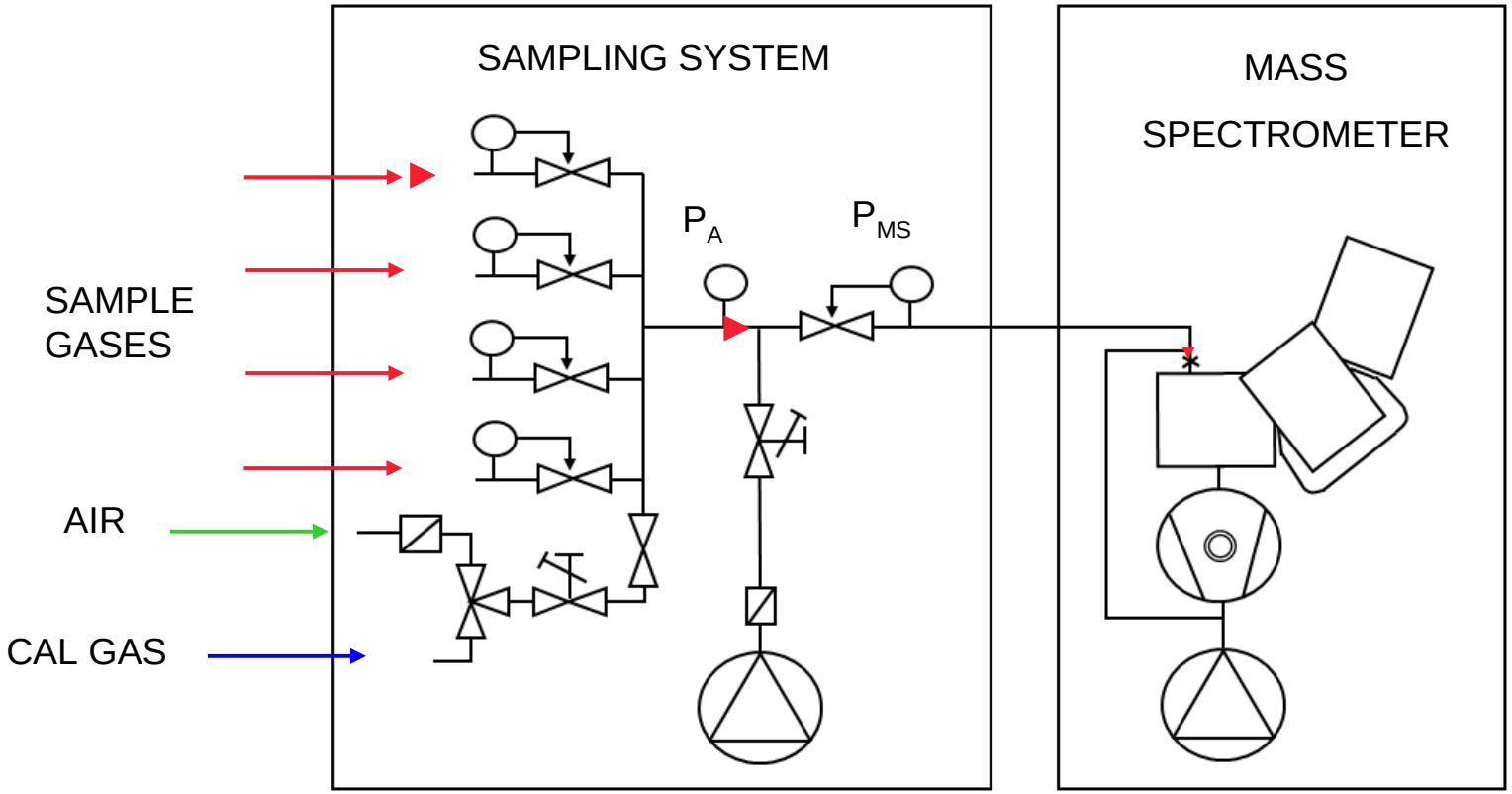
Ideally the process should be monitored directly on-line to confirm the drying is proceeding normally and enable end-point determination

Using Process MS to determine solvent drying end-point



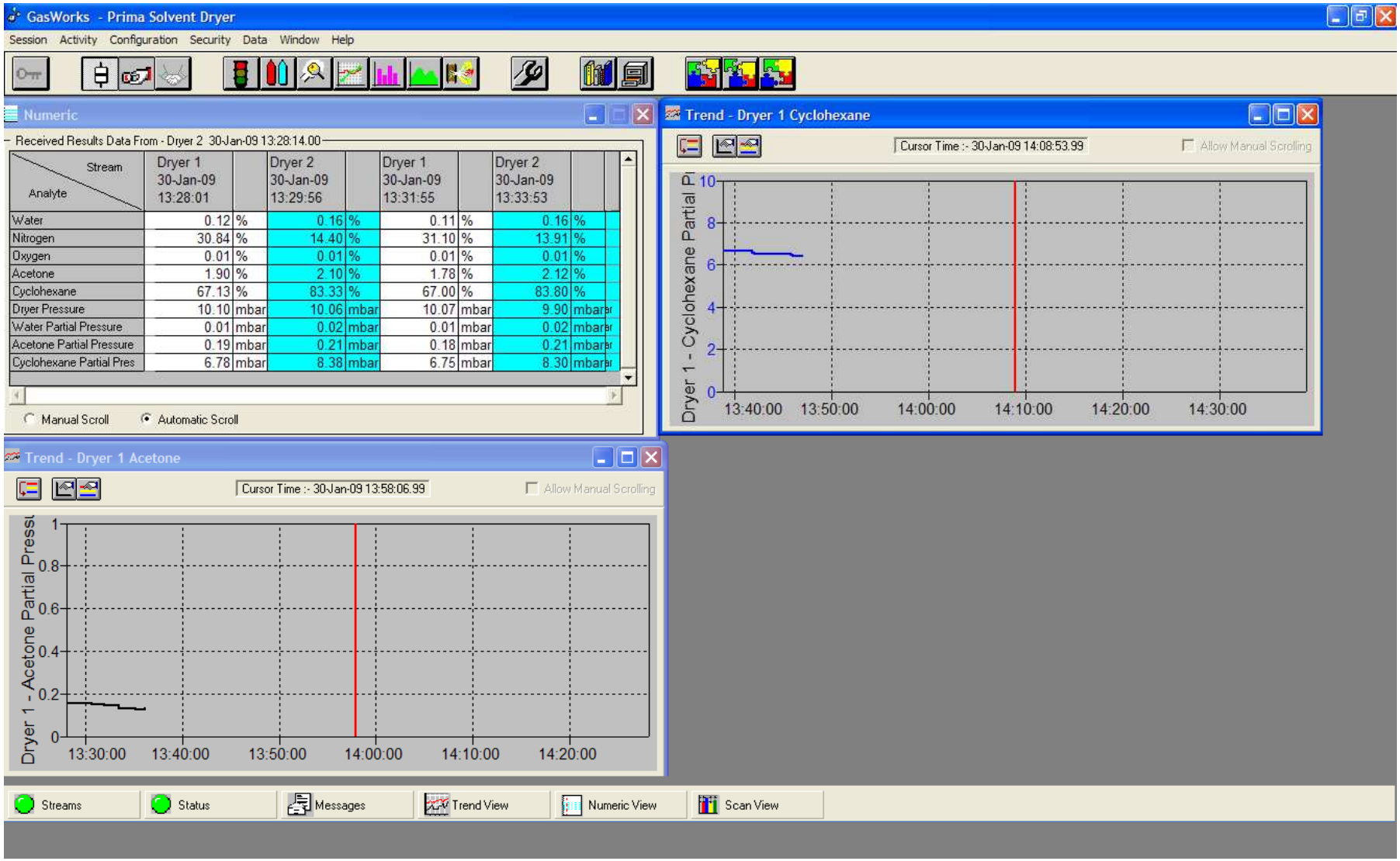
Partial Pressure of Solvent above drier is related to concentration in product material

Example System for Monitoring Multiple Dryers (at between 1 and 100 mbar) by Process MS



- Pressure switches interlock samples when pressure exceeds ~120 mbar
- P_A is absolute pressure gauge range 0 – 120 mbar
- P_{MS} is absolute pressure gauge controlled via valve typically at 0.5 mbar
- When no sample flowing, uses filtered air to prevent back flow of oil from rotary pump

Real Time Numeric and Trend Displays of Partial Pressures of Solvents in Drier



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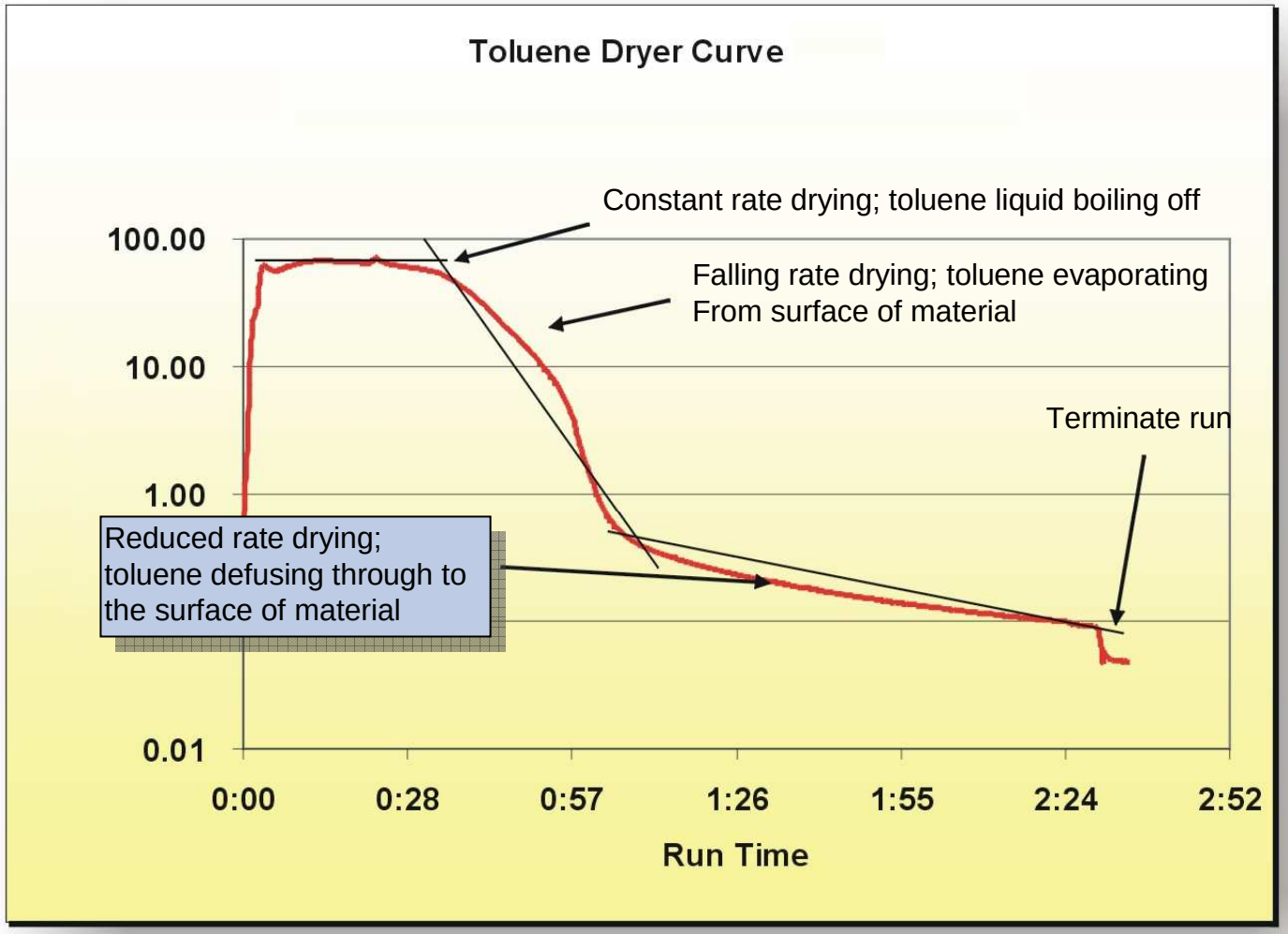
Example Vacuum Dryer Data



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Summary

- Variable Vacuum Samples require dynamic adjustment to maintain optimum performance (sensitivity and accuracy)
- Special Inlet Systems have been designed for:-
  - Secondary Steel Production (VOD & RH)
  - Pharmaceutical Powder Vacuum Drying