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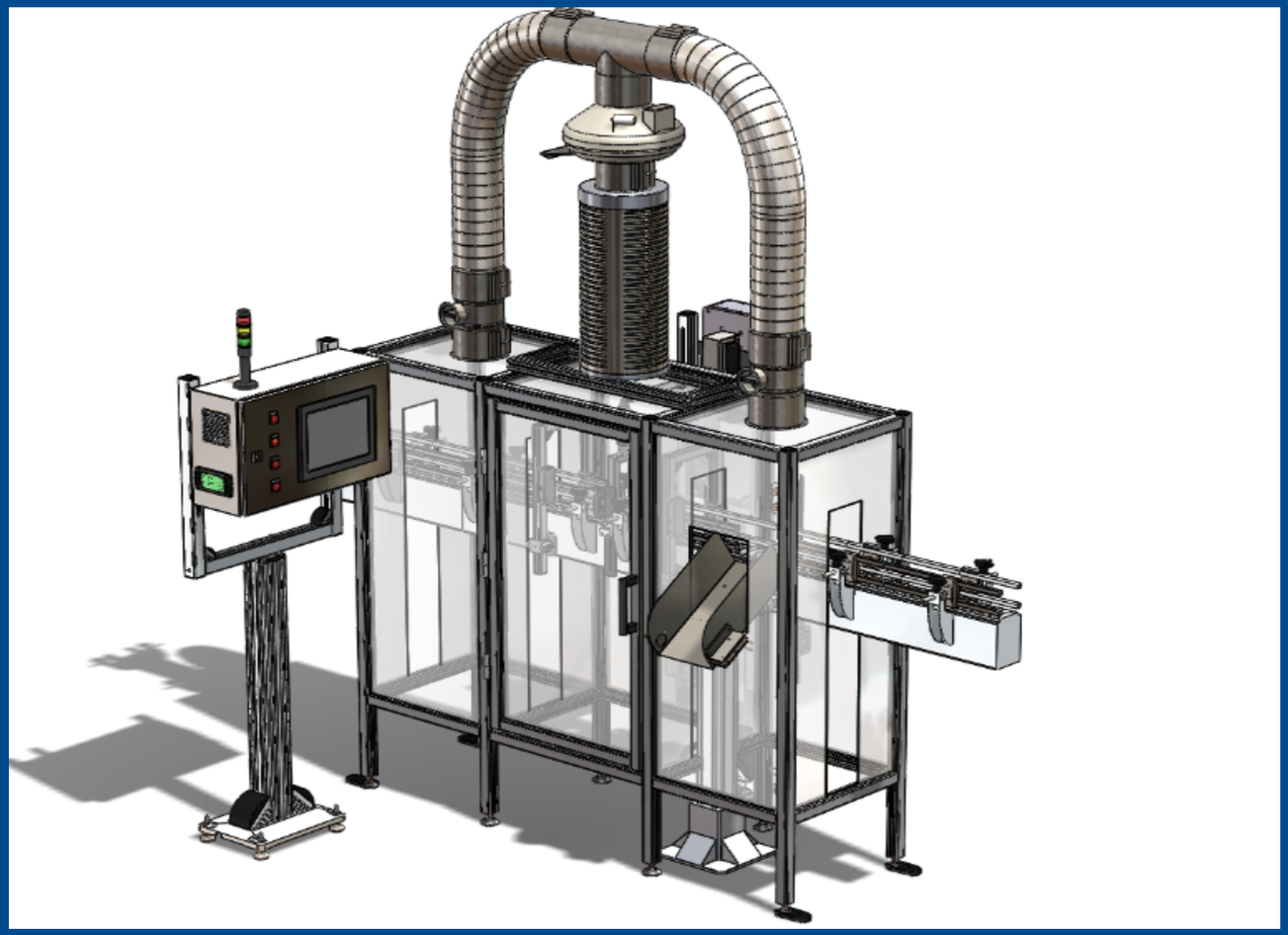
Polski Związek Pracodawców Przemysłu Aerozolowego
Polish Union Of Employers Of Aerosol Industry

Aerosol Micro Leak Detection Technology

Cascade™

The Use of Micro Leak Detection and Analysis in Process Improvement



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Inline Detection

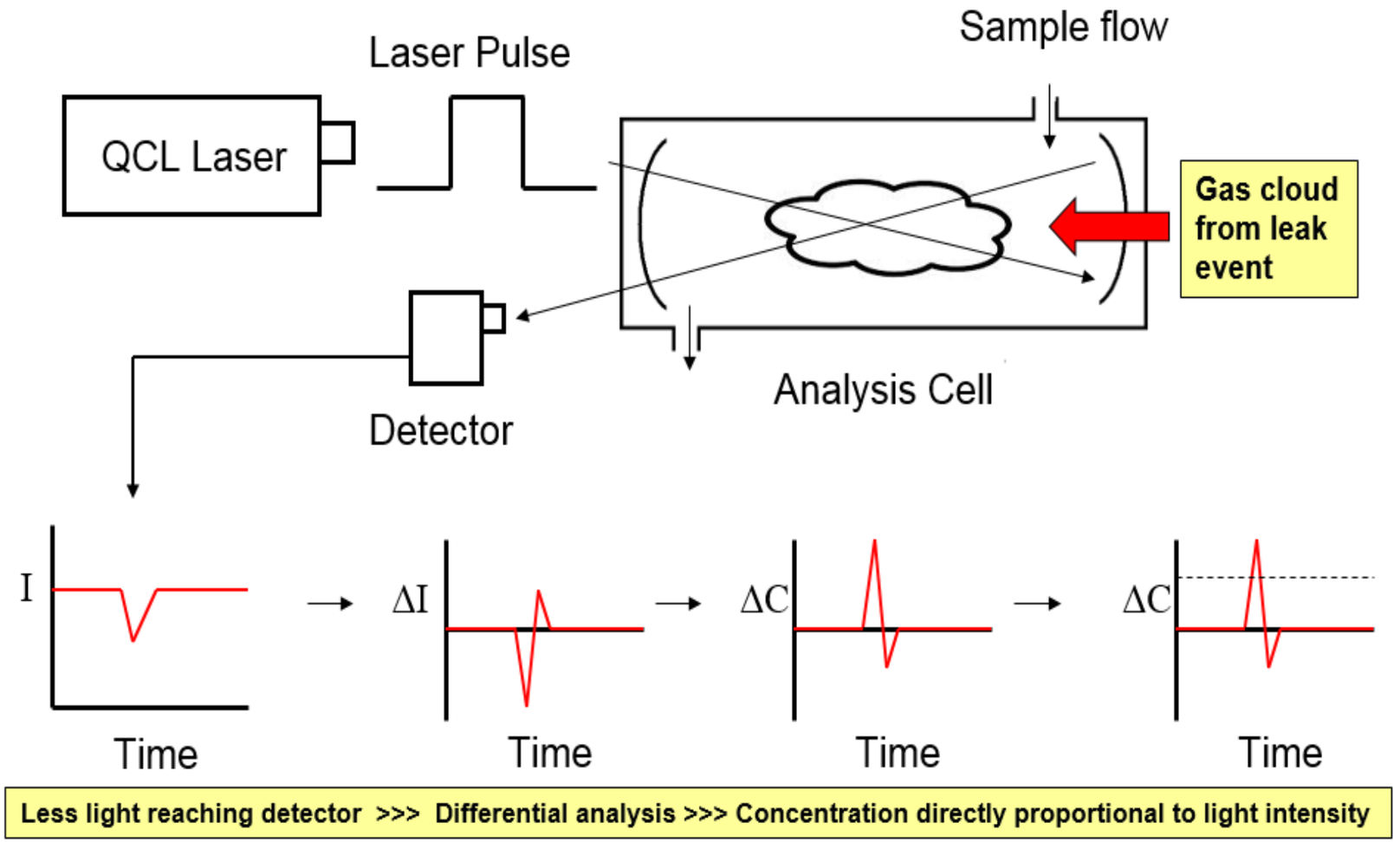
Inline Leak Detection

- Inline systems are simple detectors
- They detect the propellant leaking from a container and compare this level to a pre-set threshold.
- If this threshold is exceeded, the can will be rejected.
- This can give us valuable information on batch performance
- Inline systems identify leaks, but do not quantify them.
- So, how do they work….

Inline Systems Detect, Not Measure.

Leak Detection – How it works

- Cans pass under an archway where a vacuum is being drawn.
- As a leaking can passes the leaking propellant is drawn into a sample cell
- Some of the laser light is absorbed by the gas
- This reduction in laser intensity is compared to a threshold



Cascade™ CT2211 Leak Detector

"Used on aerosol filling production lines in conjunction with a control and reject mechanism to manage the safe removal of faulty cans at high speed."

- Propellant Capability
  - LPG, R134a, CO_2, N_2O, DME, HFO and more
- Optimised accumulation tunnel for sample capture
- Sensitivity: Meets ADR & ADD requirements for both use with a waterbath and waterbath alternative
- Rejects leaking cans in real time from production line for further analysis
- Tests 100% of the can and 100% of the cans.
- Detection tells us we have a problem – we need to know more…..

Aerosol Micro Leak Detection Technology

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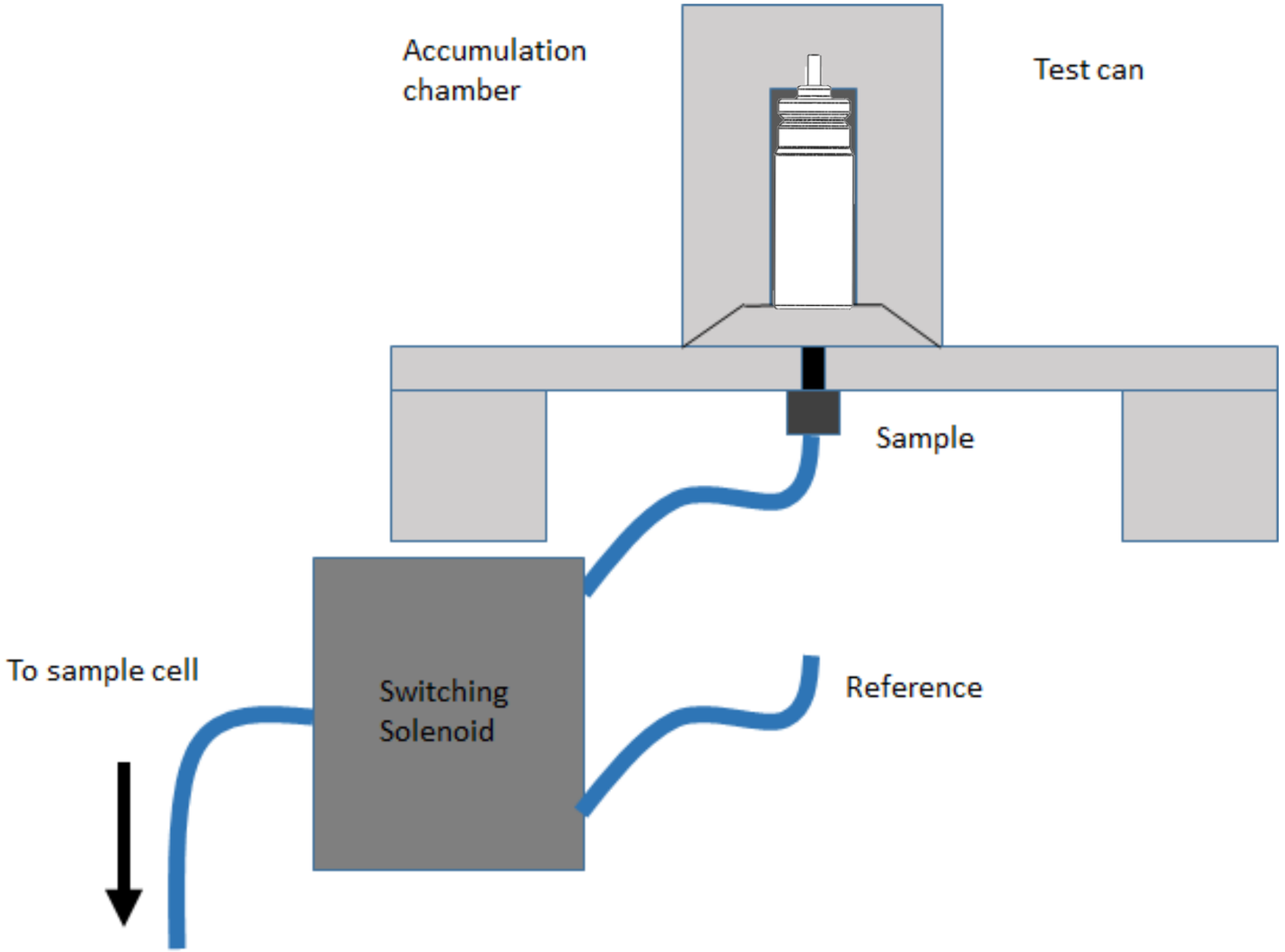
Offline Analysis

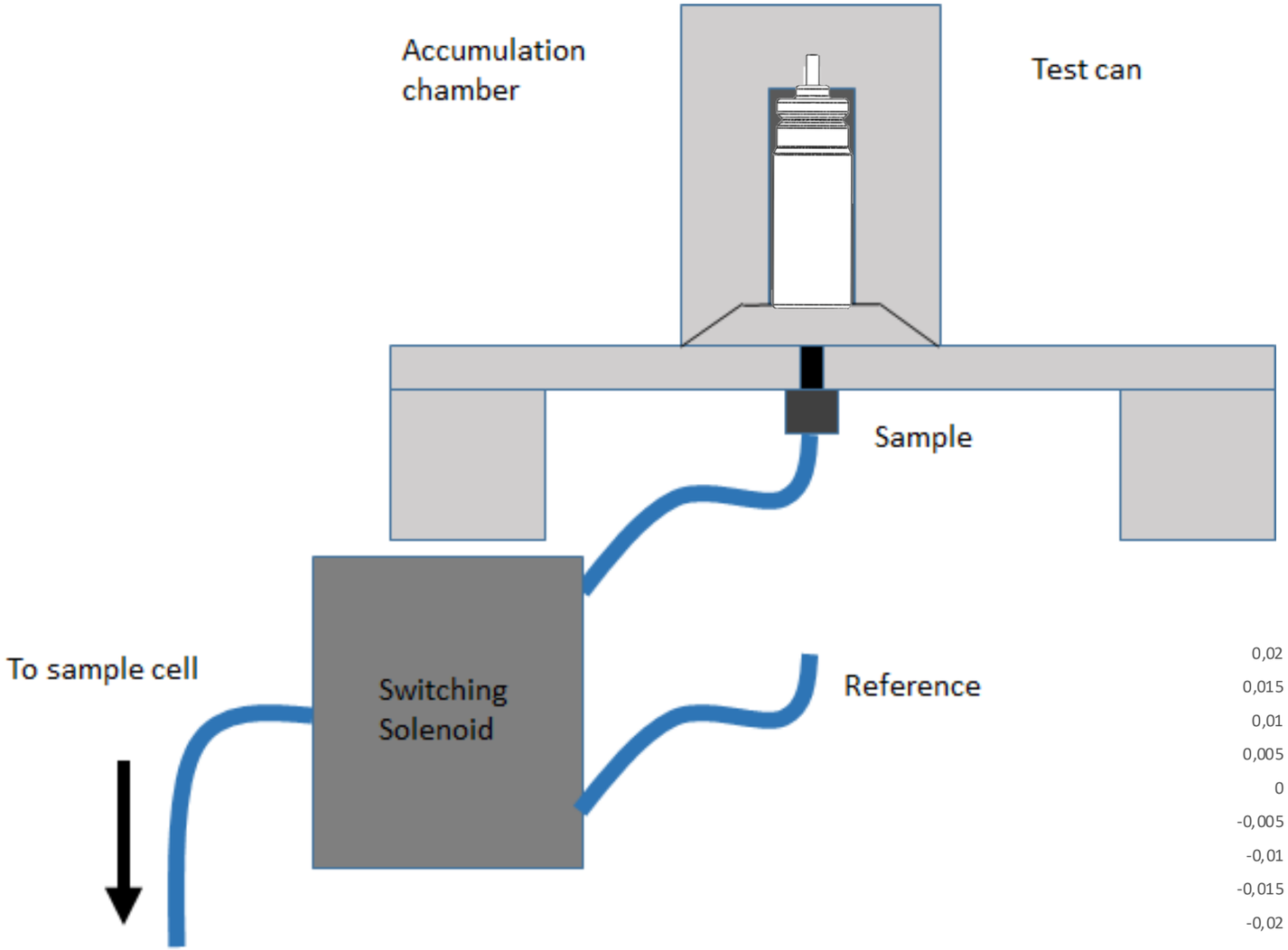
Applications

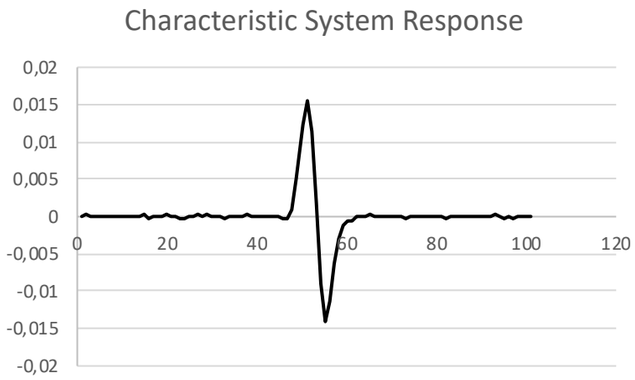
- Leak Source Identification
  - Stem
  - Crimp
  - Can Body
  - Whole Can
- Leak Kinetic
  - Leak Rate Over Time
  - Crimp Optimization Studies
  - Valve Sealing Characteristics
- New Component Evaluation
  - Evaluation of Spray through caps
  - Top Load Testing

How Does it Work?

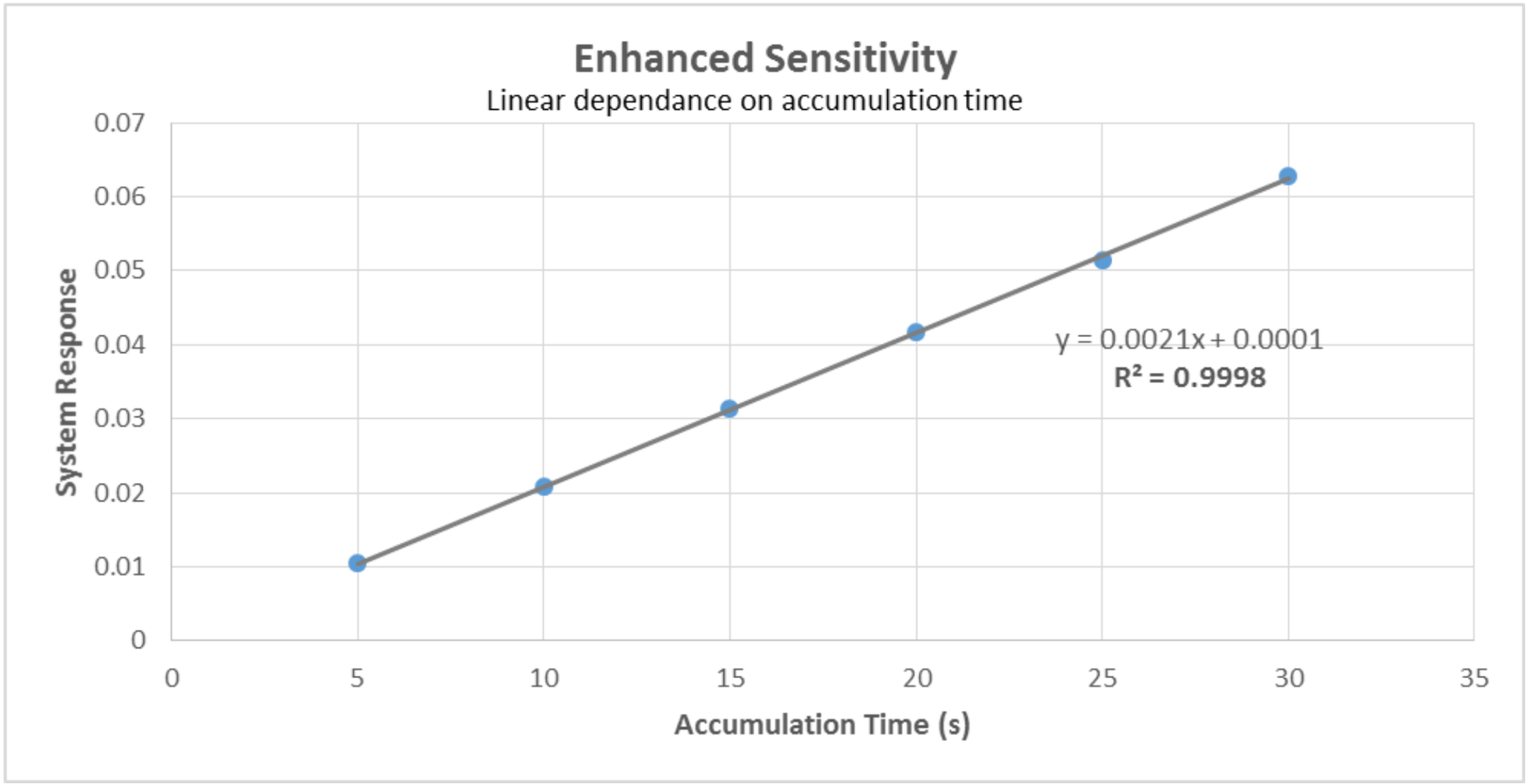


How Does it Work?





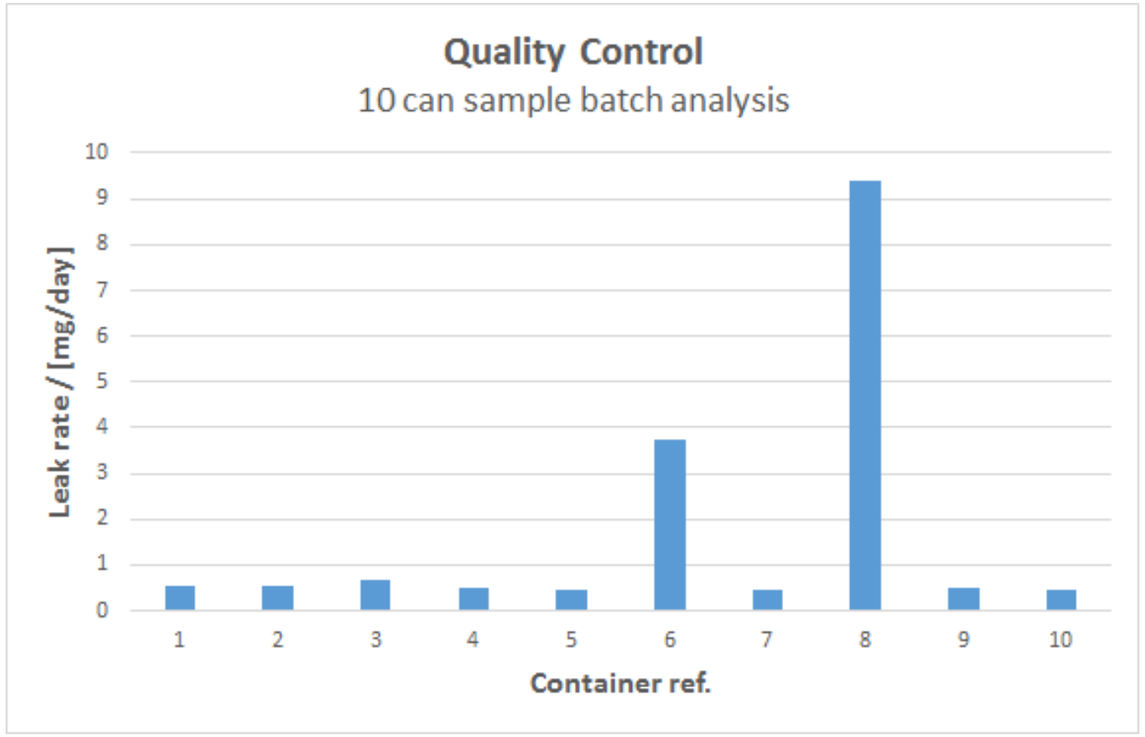
Scalable Sensitivity w/ Accumulation Time



- Linear response to accumulation time
- Scalable sensitivity

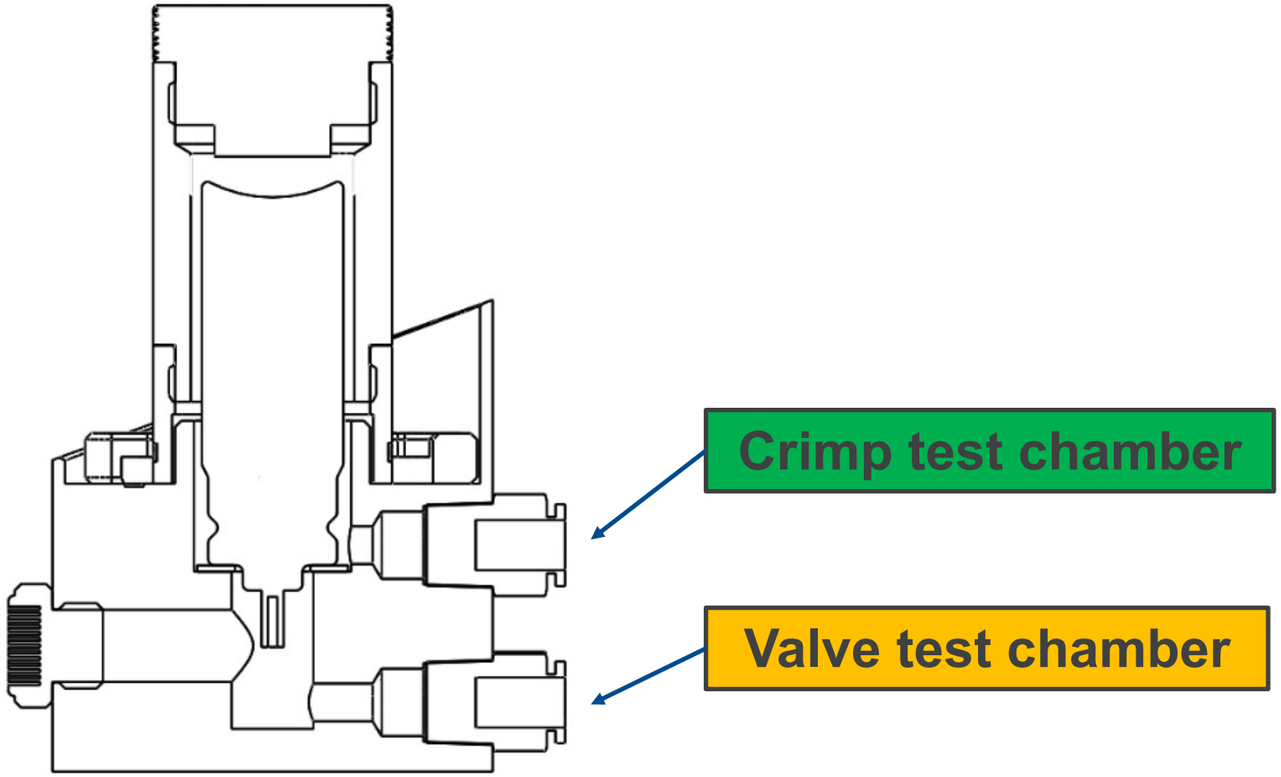
Simple Leak Detection

- Fast QA testing – total test time < 2 mins
- Traditional methods 48 – 72 hours



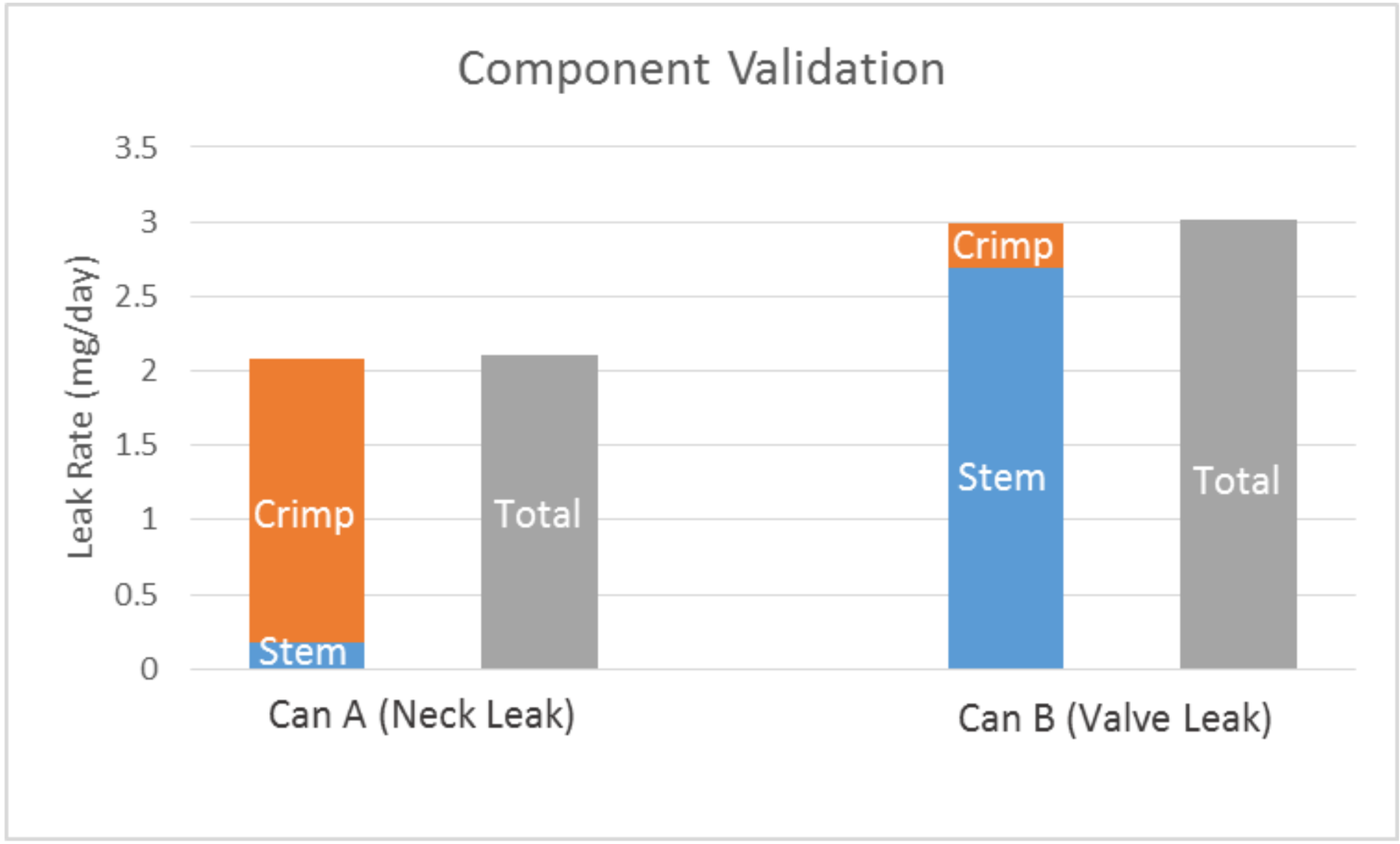
Leak Detection Is Only Part Of The Picture

Multi-chamber Design for Process Fault Identification



Patent pending sampling head – identifies the leak at the source
Automated switching

An Enabling Technology



We need to know where the canister is leaking & how much

Understanding the Leak Kinetic

Leakage is a Complex Process

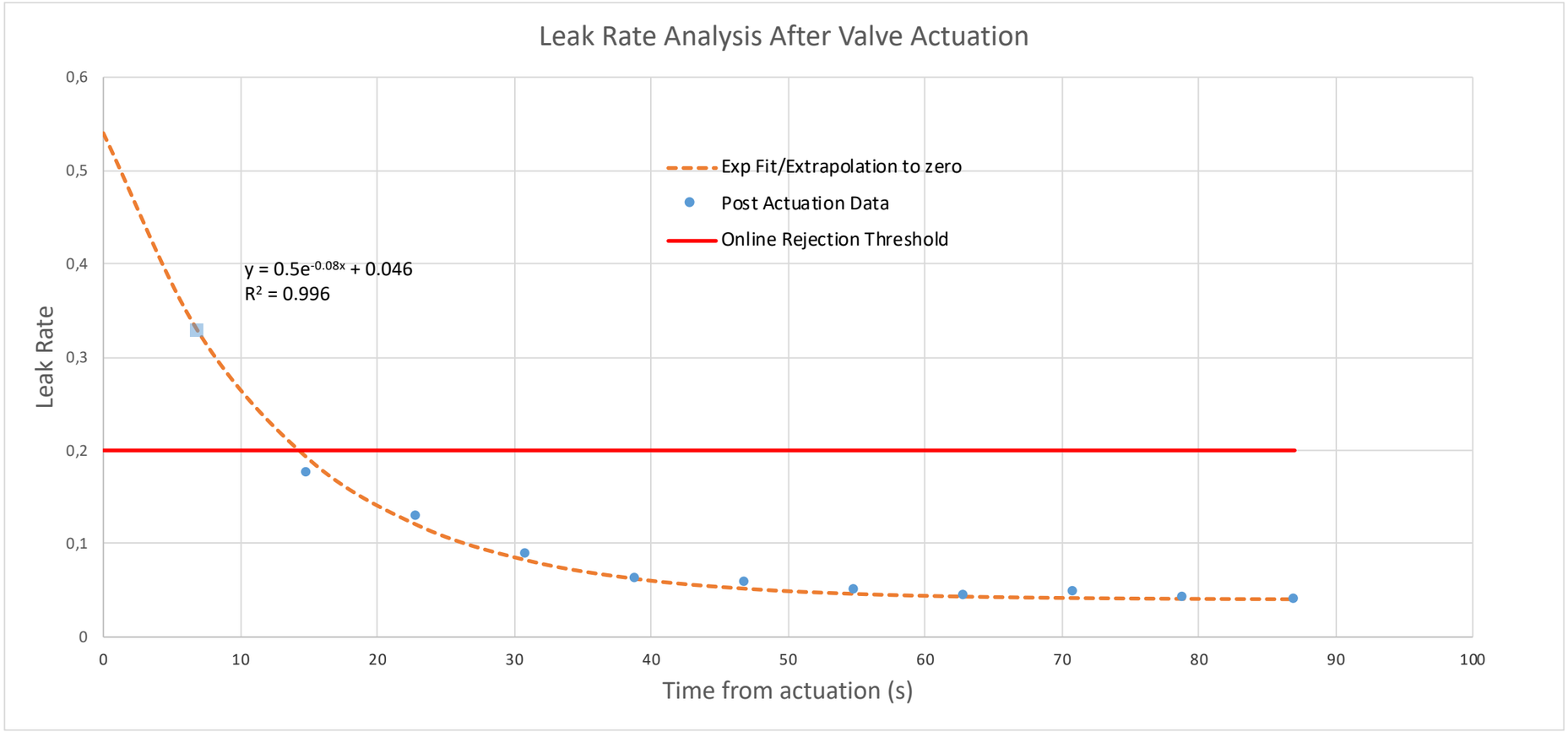
- Assembling a aerosol is a complex process and relies on a number of different components, materials and mechanical processes all performing in the way they were intended.
- Crimping, gasket material and interaction and product filling all can create leak paths.
- As the materials stabilise, the leak rate can change.

Examples of Leak Scenarios

- Immediately the can is filled the gasket may not be fully seated and has not yet swollen. This can lead to a higher leak rate - **Scenario One**
- Shot testing can introduce powder into the valve gasket area and create a leak path – **Scenario Two**
- The process and components are completely optimum and the pack is unaffected by actuation/gasket swell etc – **Scenario Three.**
- The patient does not shake the container correctly during use and high powder loadings build up on the sealing faces and can begin to leak – **Scenario Four.**

Leakage is Complex

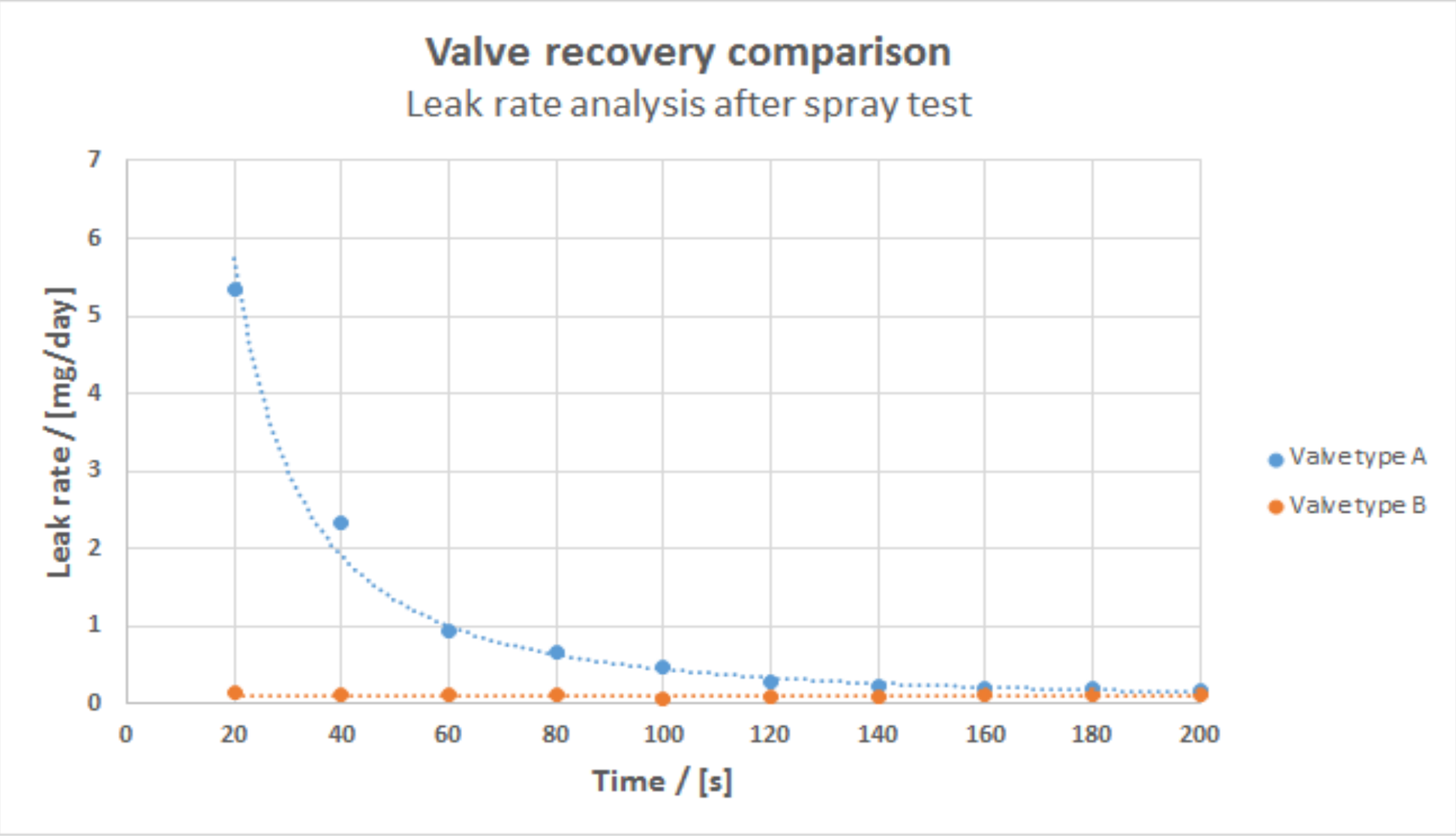
Leak Kinetics – Process Optimisation



- Valve shows slow sealing characteristics
- Propellant is lost on each actuation
- Critical if compressed gasses are used.

Instant Measurement Shows Never Before Seen Detail

Leak Kinetics



- Valves of a different type perform differently

Not All Valves Are Created Equal

Leak Rate Study – Post Filling

- A more detailed study was carried out to investigate further

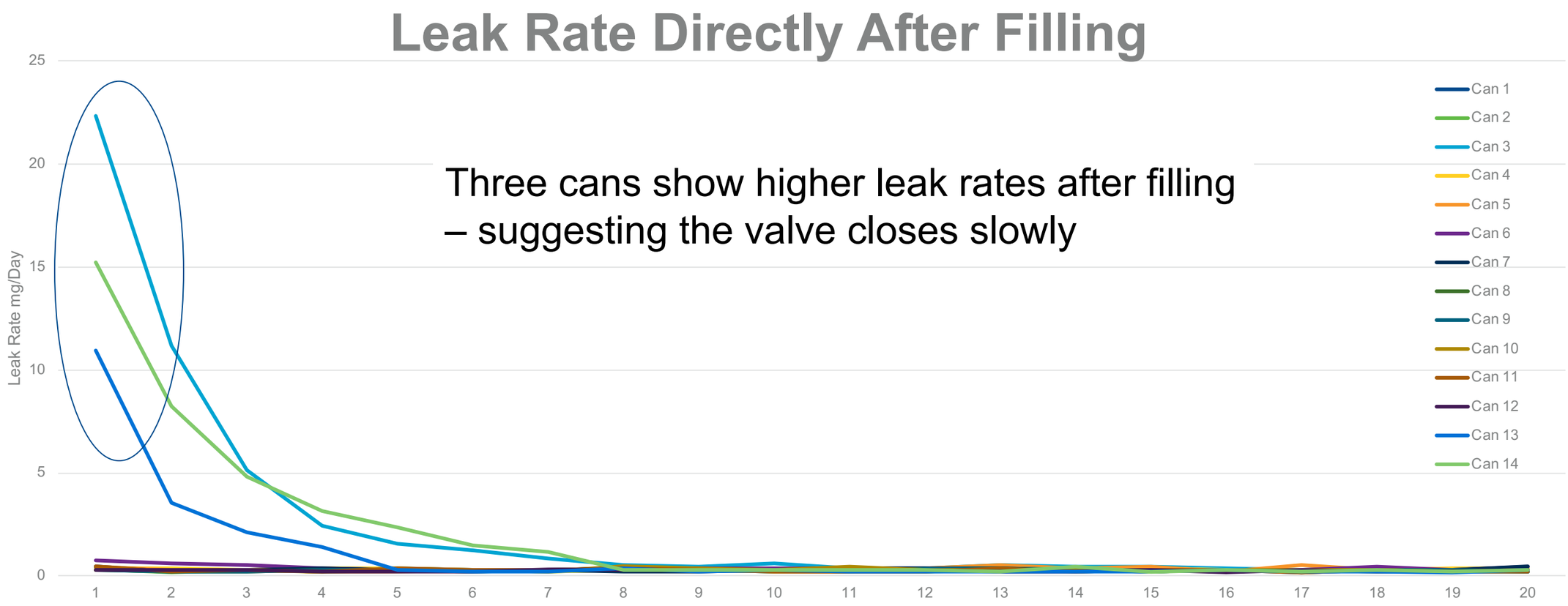
- **Protocol**
  - Propellant: 134a
  - Same valve from same manufacturer and batch.
  - Measurements taken as quickly as possible after filling
  - Accumulation time: 20s
  - Measurement units: mg/day.
- **Results**
  - Both Special Cause and Normal Variation seen.

Normal Variation:
All processes have common cause variation. This variation, also known as noise, is a normal part of any process. It demonstrates the true capability of a process.

Special Cause Variation:
This variation is not normal to the process. It is the result of exceptions in the process environment.

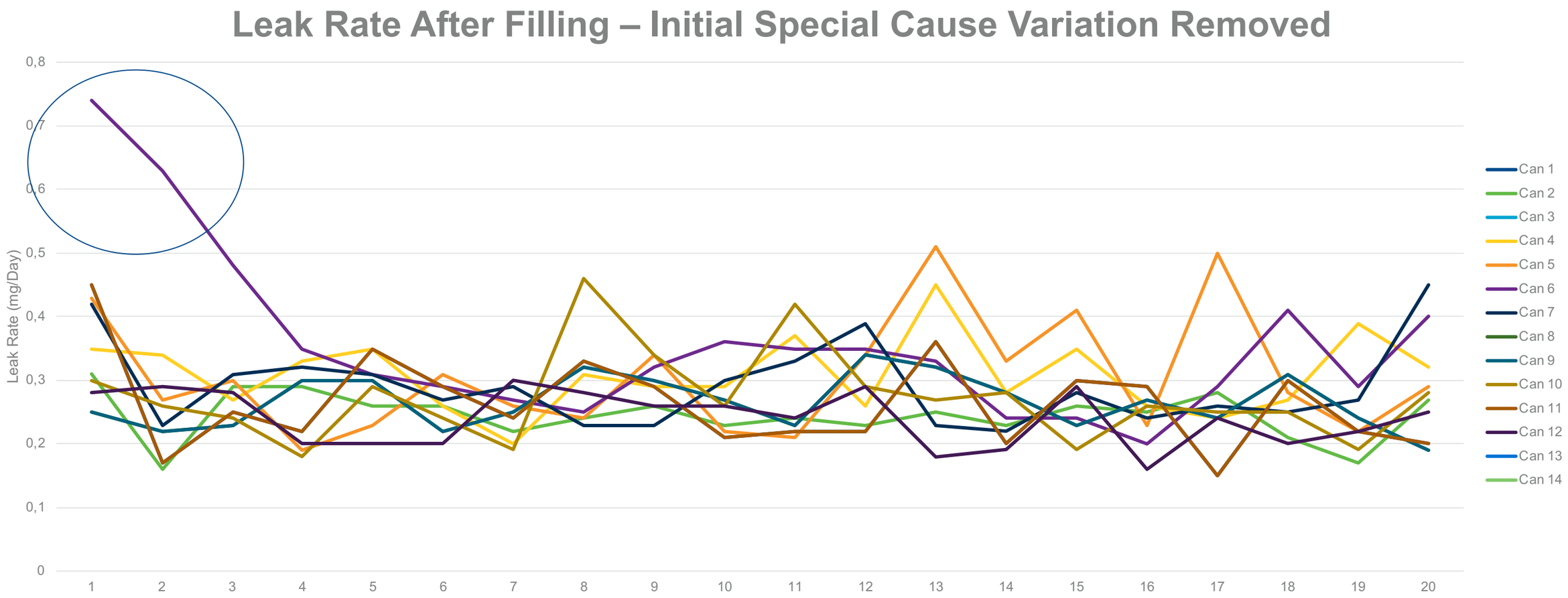
Real Study Data Shows A Level Of Detail Not Previously Possible.

Special Cause Variation



3 Out Of 14 Showed Anomalous Behaviour

Same Data – Initial Special Cause Removed



Stripping Out Special Cause Variation Shows Hidden Detail

SUMMARY

- The CT4404 is a robust and easy to use system to determine leak rate in Aerosols
- It can measure leakage from both crimp and stem, or simply whole can.
- Customisable sample heads mean specific studies can be accommodated – for example: Studying of the effect of top loading on spray through caps.
- Sensitivity can be varied depending on accumulation time.
- Butane/Propane, DME, 134a, Carbon Dioxide can be measured.
- High sensitivity mean very low leak rates can be monitored and since the measurement typically takes 20s, real-time leak kinetics can be studied.
- Process studies are possible – or example gasket selection can be confirmed with real-time leak rate after filling, monitoring the effect of gasket selection on critical products, such as those with low propellant levels.

Detailled Leak Characterisation Is Now Possible

Questions?

Validation

- Equipment required:
  - Cylinder of known, certified concentration for gas of interest
  - Calibrated Gas Divider with regulator
  - Clean compressed air supply
  - CT4404 Leak Analyser
  - Dummy Canister – for use if Dual chamber system is used.

- Test Procedure
  - Connect cylinder to gas divider input
  - Connect the Gas Divider output to the validation port of the CT4404
  - Turn on cylinder regulator to xx bar
  - Turn on Air supply and gas divider
  - Set dilution required on gas divider
  - Begin measuring with CT4404 and monitor until a steady state reading is obtained.
  - Check reading obtained by CTT4404 matches that of the gas divider e.g. Cylinder concentration = 100ppm, dilution 50%, reading should be 50ppm+/-5%
  - Repeat at multiple dilution factors.