O_2 Fingerprinting in Pasteurised Beverage Products Using Headspace Gas Analysis (Orbisphere 6110)

Objective

This report aims to evaluate the use of the Orbisphere 6110 for headspace gas analysis in pasteurised carbonated beverage packaging. The specific goal is to develop an O_2 fingerprinting approach to identify post-pasteurisation oxygen ingress. A particular challenge is that dissolved oxygen can react with the beer matrix over time, rendering direct O_2 measurement increasingly unreliable. Therefore, a derived metric—**residual gas volume** adjusted for CO_2 purity—can be used to infer the **historical presence and consumption** of oxygen over storage duration. This enables better root cause analysis and quality decision-making across the product lifecycle.

Background

The Problem of Oxygen Ingress

Oxygen ingress after pasteurisation is a critical concern in beer packaging. Even trace levels of O_2 can:

- Degrade flavour and aroma compounds (oxidative staling)
- Reduce shelf life
- React with polyphenols, proteins, and other sensitive compounds
- Microbiological instability
- Loss of carbonation

After pasteurisation, bottles are sealed to preserve a CO_2-rich environment (>99% CO_2 in headspace). However, oxygen ingress may occur due to

- Cap/crown sealing issues
- Leaks exacerbated by thermal expansion during pasteurisation
- Material permeability
- Handling and process variability

may allow atmospheric gas ingress, particularly oxygen and nitrogen.

Any oxygen ingress that occurs post-pasteurisation is critical because the product can no longer be thermally treated to mitigate its effects.

Why Traditional O_2 Monitoring Falls Short

While direct measurement of headspace or dissolved O_2 can be effective shortly after packaging, it becomes increasingly misleading over time:

- Oxygen reacts chemically with beer components (e.g., aldehyde formation, polymerisation)
- These reactions consume the O_2, reducing detectable levels
- This makes it difficult to determine whether ingress occurred weeks or months earlier

The **Orbisphere 6110** is a high-precision instrument designed for analysis of headspace gas composition in beverage packaging. Unlike traditional TPO or dissolved oxygen methods, headspace analysis:

- Directly assesses the gaseous environment above the liquid
- Identifies deviations in gas composition attributable to post-process ingress
- Allows process engineers and quality teams to pinpoint the timing and mechanism of contamination

A New Approach: Residual Gas Fingerprinting

- Measures total gas volume and composition in the headspace
- Compares actual CO_2 purity against expected values
- Uses a formula to estimate non-CO_2 gas volume (i.e., foreign gas), which indirectly reflects historical oxygen ingress

This method can detect signs of **previous oxygen ingress even after the oxygen has reacted,** offering a powerful tool for process monitoring, QA diagnostics, and long-term storage evaluation.

Method

Equipment & Sample Handling

Device: Orbisphere 6110 Headspace Gas Analyzer
Product Type: Pasteurised beer in glass bottles

Test Protocol

1. Analyse headspace gas composition immediately after pasteurisation.
2. Capture CO_2%, O_2%, and pressure data.
3. Apply foreign gas formula.
4. Evaluate test repeatability on identical batch samples.

Orbisphere 6110 Total O_2/CO_2 Package Analyzer & Installation Kit

Results and Analysis

Results

Package Age	TPO (ppb)	CO2 (True)	Vol. HS	Residual Gas (mL)	% Purity CO2
1 day	52.69	5.37	18.2	2.2	98.3
1 day	65..9	5.35	18.78	2.5	98.2
1 week	73.89	5.39	18.85	4	97.4
1 week	63.26	5.41	18.22	3.9	97.6
2 weeks	119.74	5.34	18.75	4.4	97.2
2 weeks	92.59	5.33	18.54	4.5	97.1
3 weeks	108.53	5.41	18.09	3.7	97.6
3 weeks	83.18	5.45	17.54	3.7	97.5
4 weeks	93.84	5.33	17.53	3.7	97.5
4 weeks	82.45	5.35	17.2	3.9	97.4
3 months	87.23	5.33	18.36	4.6	97.2
3 months	58.27	5.32	20.13	4.2	97.5
3 months	133.51	5.44	17.039	4.8	96.9
3 months	115.24	5.43	19.51	4.9	97.2
6 months	95.48	5.09	18.96	5.9	96.5
6 months	227.7	5.05	18.48	6.4	96.1
12 months	440.45	4.87	21.47	6	96.7
12 months	63.06	5.08	17.85	4.9	96.8

Analysis

Key Observations by Package Age

Age	Avg TPO (ppb)	Avg. CO_2 Purity (%)	Avg. Residual Gas (mL)
1 day	59.30	98.25	2.35
1 week	68.58	97.50	3.95
2 weeks	106.17	97.15	4.45
3 weeks	95.86	97.55	3.70
4 weeks	88.15	97.45	3.80
3 months	98.56	97.20	4.63
6 months	161.59	96.30	6.15
12 months	251.76	96.75	5.45

Trends & Interpretations

TPO (Total Package Oxygen):

- Increases significantly with time. Starting from ~59 ppb at 1 day and reaching >250 ppb at 12 months.
- Notable jumps at 2 weeks, 6 months, and 12 months, suggesting progressive oxygen ingress over time.

CO_2 Purity:

- Starts at ~98.2% and steadily declines to ~96.7% by 12 months.
- Indicates gradual ingress of foreign gas, likely air (O_2 and N_2).
- Drop is modest (~1.5%), but consistent, supporting the ingress hypothesis.

Residual Gas Volume:

- Increases from ~2.35 mL at 1 day to ~6.15 mL at 6 months Suggests either:
  - More headspace gas due to liquid CO_2 loss or
  - Introduction of foreign gas into the package
  - Correlates with increasing TPO and decreasing CO_2 purity.

Correlation Insights

Age	Avg. TPO (ppb)	Avg. CO_2 Purity (%)
TPO vs. Age	+0.91	Strong positive - oxygen ingress over time
CO_2 Purity vs. Age	–0.86	Strong negative - loss of CO_2 purity with time
Residual Gas vs. Age	+0.87	Strong positive - headspace gas increases as ingress occurs

These strong correlations support the foreign gas ingress hypothesis, likely due to:

- Cap permeability or microleakage
- Thermal cycling stresses on crown/cap integrity
- Storage conditions over time

Conclusions

- O_2 ingress increases significantly over time, confirming the value of early and repeated headspace analysis.
- CO_2 purity drops consistently, validating its use as a diagnostic parameter.
- Residual gas volume increases are indicative of either CO_2 loss or external gas ingress.
- The Orbisphere 6110 is well-suited for tracking these parameters in a non-destructive and repeatable manner.
- Apply SPC methods to headspace data across aging studies for predictive quality management.

Recommendations

- Establish CO_2 purity thresholds to flag early signs of ingress (e.g., alert if <97.0% within 2 weeks).
- Use TPO + CO_2 purity + residual gas volume for multi-parameter fingerprinting.
- Investigate closure integrity and bottle variability if TPO deviates early (before 2 weeks).
- Apply SPC methods to headspace data across aging studies for predictive quality management.

Discussion

The Orbisphere 6110 enables precise identification of package-level gas composition post-pasteurisation. This makes it a valuable tool for:

- Verifying seal integrity
- Diagnosing sources of oxygen ingress
- Detecting closure or filler issues
- Supporting corrective actions and preventative maintenance

Because the technique focuses on headspace changes, it provides a sharper diagnostic view than dissolved oxygen or TPO methods alone.

Recommendations

- Introduce headspace fingerprinting into routine QA for post-pasteurised lines.
- Use foreign gas formula to benchmark new packaging components or closure types.
- Integrate 6110 data with SPC systems to monitor trends and preempt issues.
- Expand testing to different beverage types and packaging formats.

Conclusion

The Orbisphere 6110 enables highly sensitive headspace gas analysis, offering a novel "O_2 fingerprinting" approach to detecting foreign gas ingress in pasteurised beverage packaging. This supports enhanced quality control, faster root cause investigations, and ultimately, more stable and consistent products.