P03

From After-the-Fact Air to Instantaneous Oxygen:

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LESSONS LEARNED TO DATE FROM IMPLEMENTING CHEMILUMINESCENT AND ELECTROCHEMICAL DISSOLVED OXYGEN MONITORING TECHNOLOGY

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ABSTRACT

For the sake of flavor stability, it is imperative to minimize exposure of beer to oxygen. In order to improve our oxygen management capabilities, chemiluminescent and electrochemical oxygen sensors were commissioned for monitoring dissolved oxygen (DO) in beer entering the filler and also for measuring DO in package. Presented herein are a collection of lessons that were learned through the commissioning of this equipment.

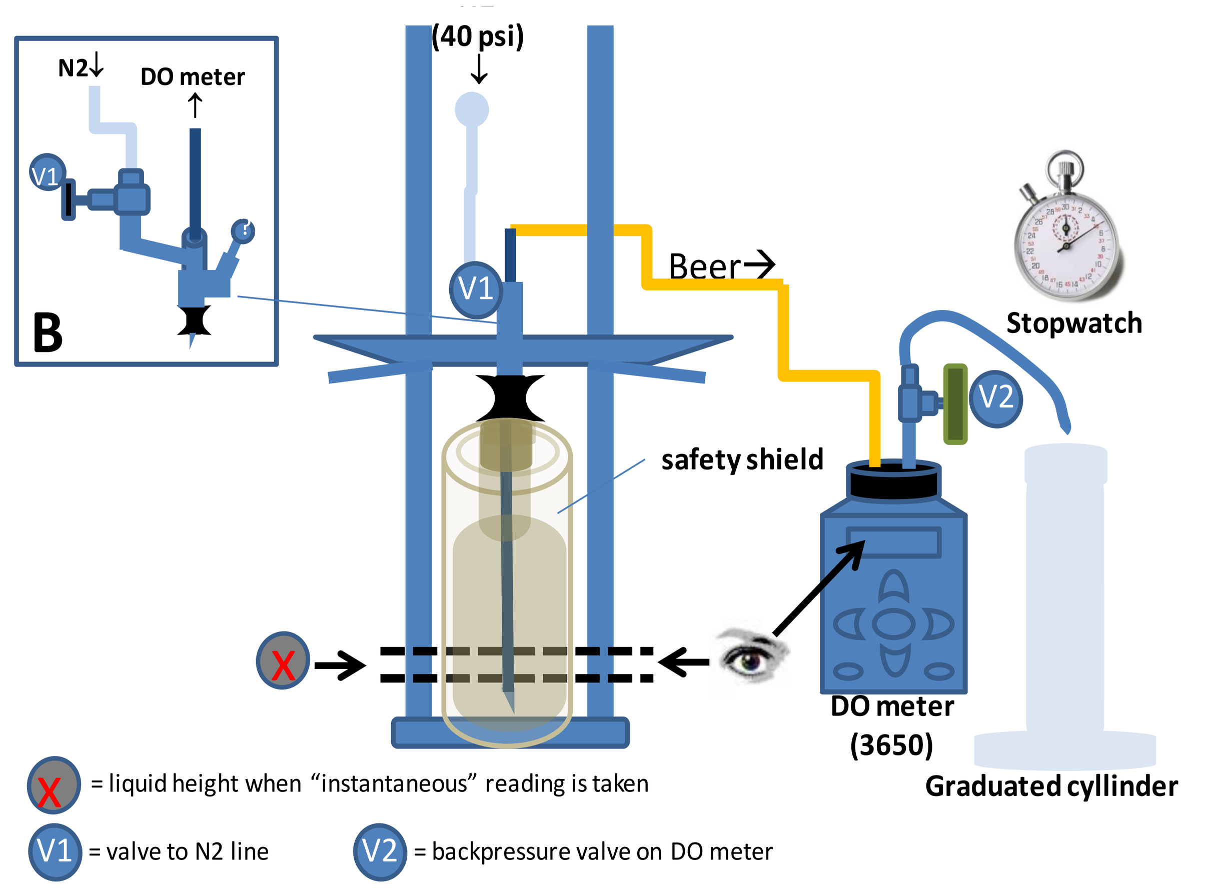


Figure 1. Schematic of first package testing system constructed in the present work

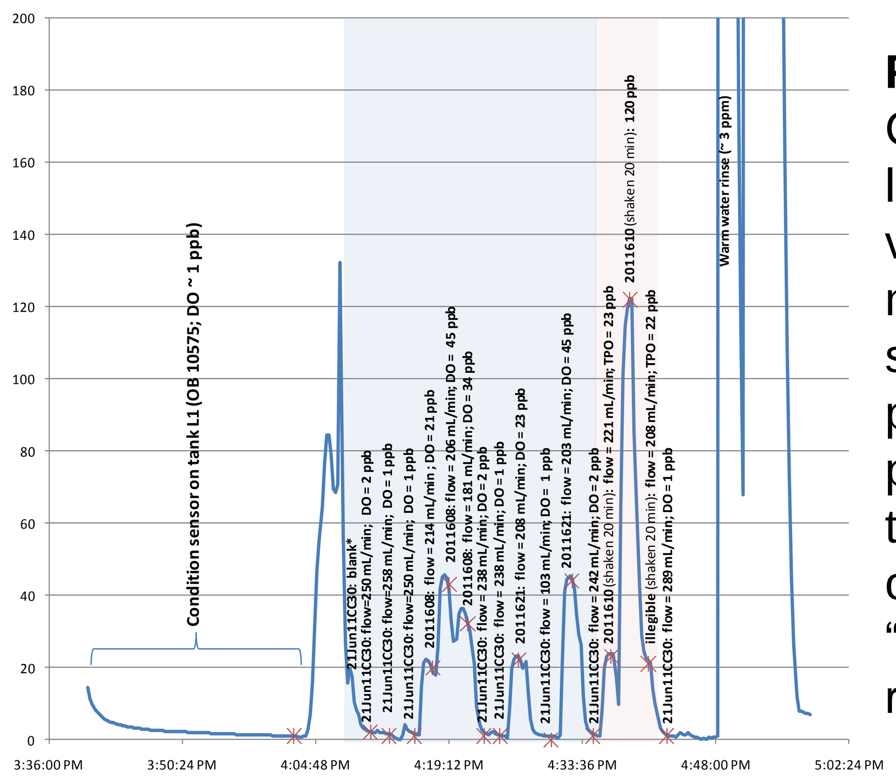
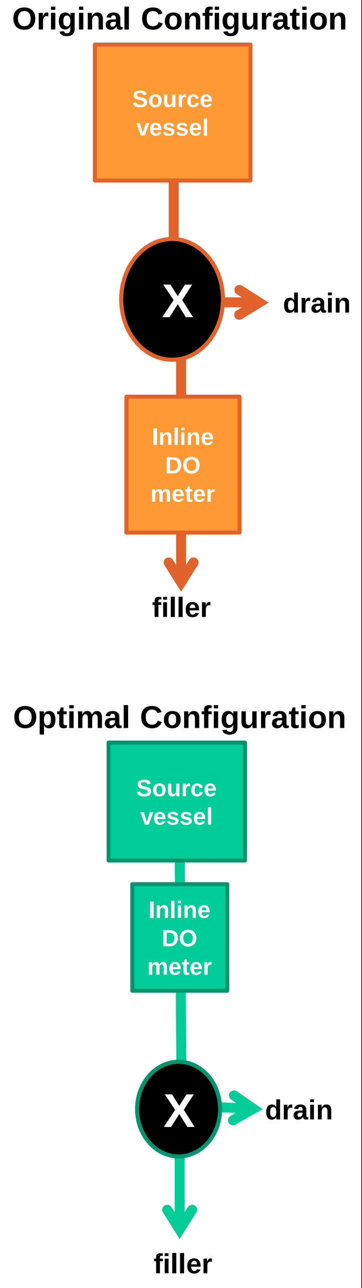


Figure 2. Continuously logged DO values from membrane sensor during preliminary package DO testing stages overlaid with "instantaneous reads."

Figure 3. Two configurations for inline DO monitor installation. The latter allows for better control of interfaces.



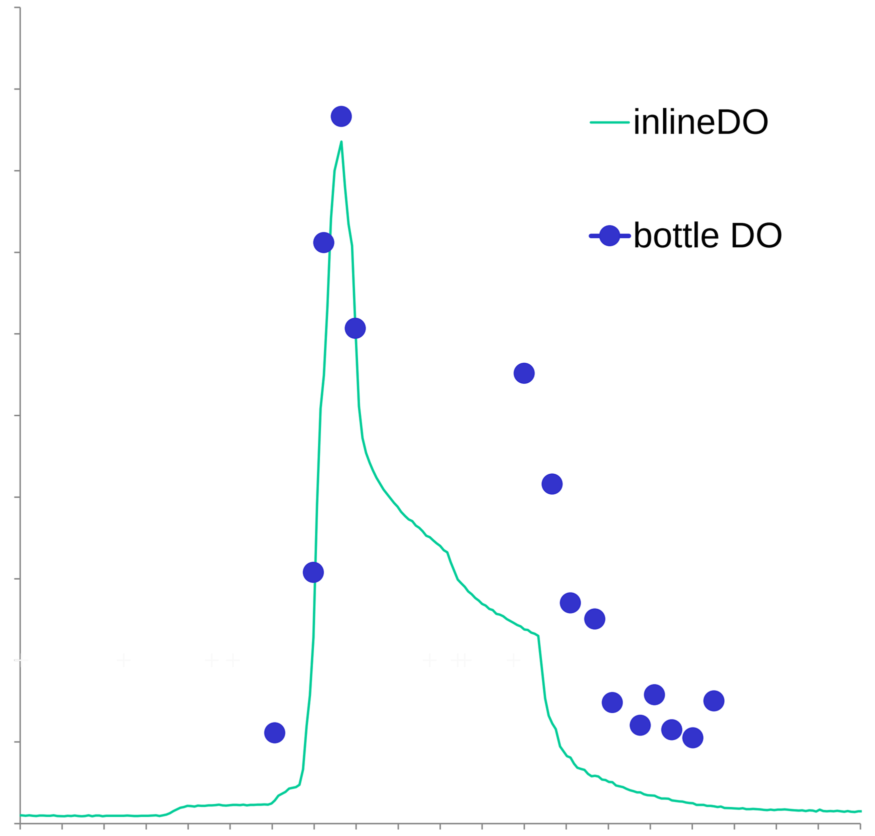


Figure 4. Relationship between Inline and package DO values.

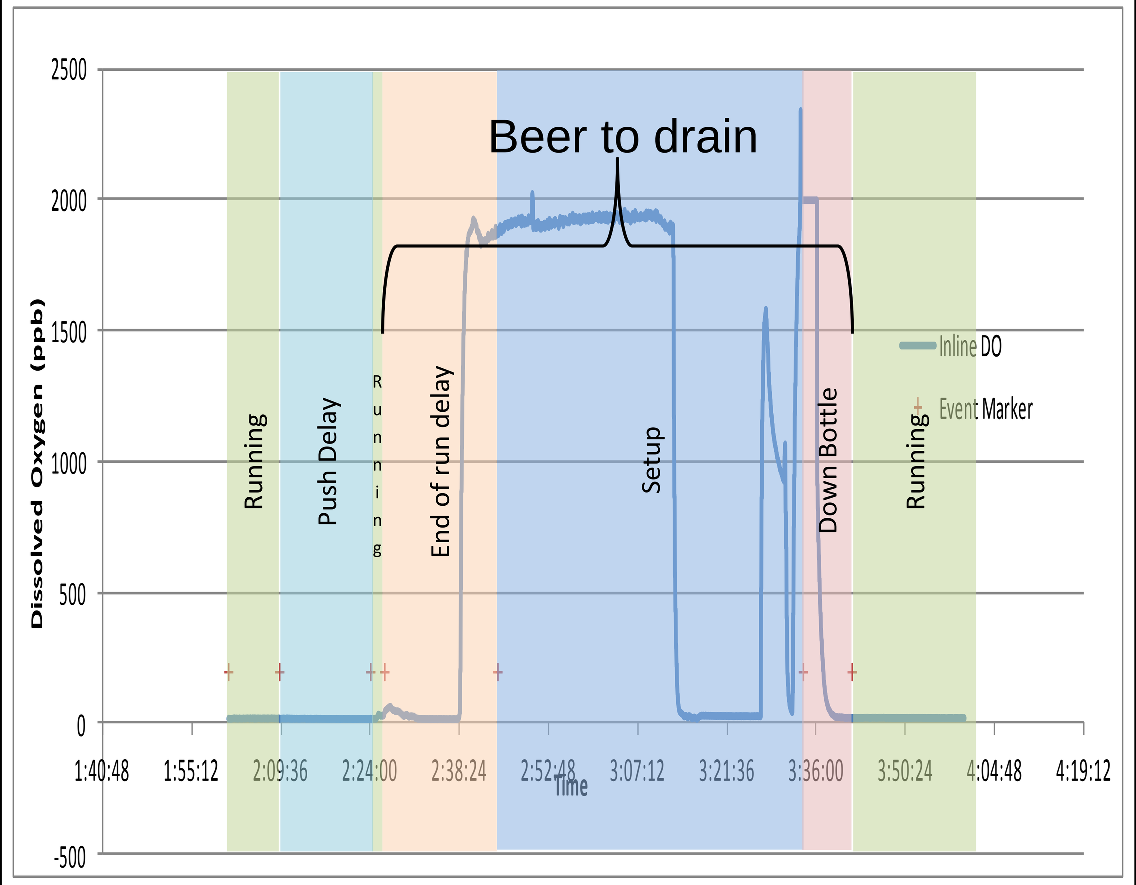


Figure 5. There are a variety of different interfaces in packaging. These interfaces must be controlled to avoid oxygen ingress into the product. Placement of drain valve after DO meter allows operator to control these interfaces.

storage temp (°C)	days stored before testing	~DO conc of high samp	~DO conc of low samp	N (total)	k (chose high)	P
22	115	224	95	5	4	0.156
22	7	290	85	6	4	0.234
36	7			6	5	0.094
22	14			6	2	0.234
36	14			6	3	0.313
22	22			7	6	0.055
36	22			7	6	0.055
22	8	162	94	8	4	0.273
22	14			9	2	0.070
22	22			8	5	0.219
22	29			10	8	0.044
22	3	1700	51	9	7	0.070
22	18			8	6	0.109
4	21			10	9	0.010
22	63			9	8	0.018
4	63			9	8	0.018
22	80			10	8	0.044
4	80			10	9	0.010
22	92			10	6	0.205
4	92			10	8	0.044
22	119			10	6	0.205
4	119			10	8	0.044

Table 1. Sensory data for IPA. Sensory information is the primary driver for optimizing action/warning limits based on DO measurements.

CONCLUSIONS

- Placement of a drain/valve relative to inline meter is crucial for empowering operators to control oxygen ingress.
- Data storage capabilities for inline meters can be quite limited (networking and external data storage solution is required).
- A package testing apparatus, which can be easily constructed by coupling a portable DO meter with a manual piercer, provides an excellent apples-to-apples comparison for troubleshooting.
- Utilize sensory to set brand based oxygen control limits.
- Cold storage (5 °C) for 3-4 weeks after crowning was found to be optimum conditions for sensory detection of differences in package oxygen.
- For an IPA, storage temperature appears to have a greater impact on shelf life than dissolved oxygen ingress during packaging.
- DO meters (inline and package testing) are powerful tools for QA as well as QC.
- Oxygen management cannot truly be obtained until it is owned by the packaging department.

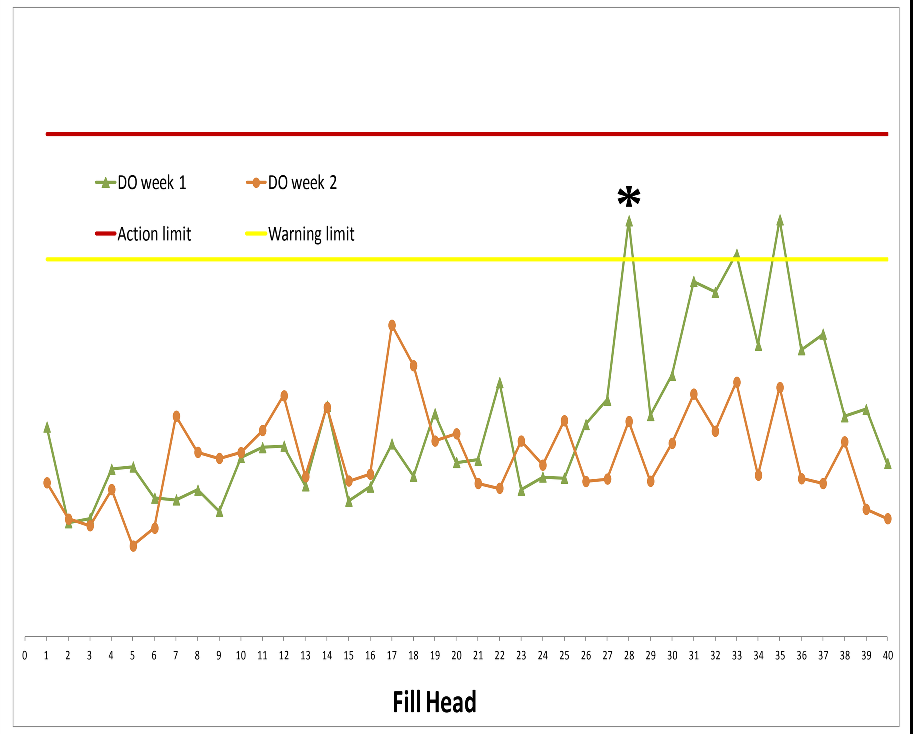
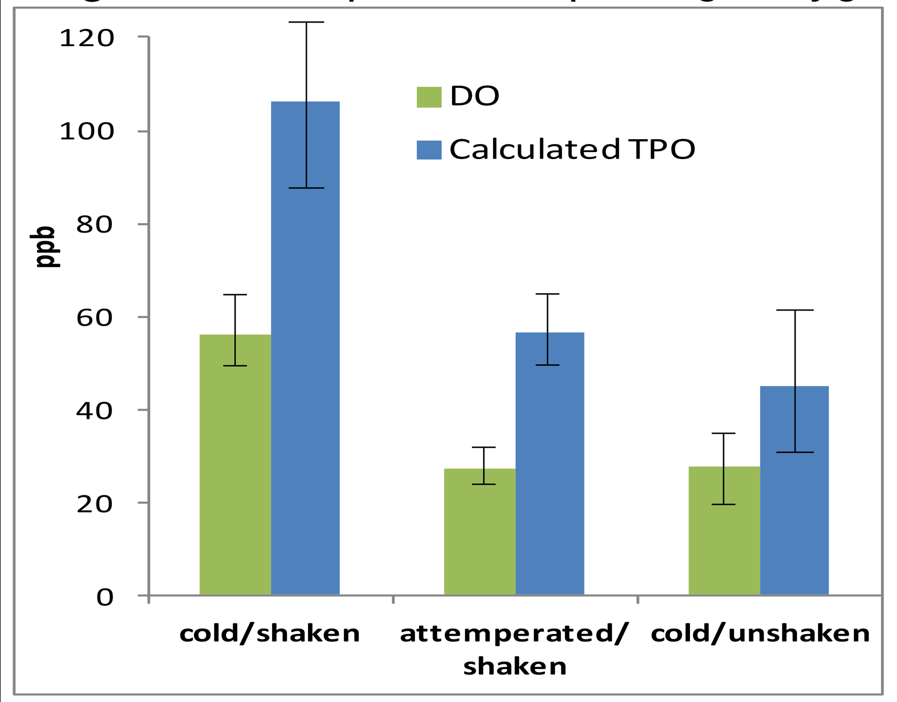


Figure 6. Package testing quickly allowed us to identify fill heads as a significant source of variation in package DO data. A routine test of all 40 fill heads was established to prioritize filler maintenance.

Figure 7. Comparison of package oxygen measurement techniques.



One of our primary objectives of the present work was to develop Package Oxygen Testing as a resource for our bottling team for real-time decision making. All internal "go/no-go" action limits and a **majority of the development work presented herein is based on measurements of cold, unshaken packages containing live yeast** taken off the bottling line immediately after crowning.

$$TPO = \frac{dO2\ (mg/L)[1 + (mol.\ Wt.\ O2)(1{,}000\ mL/L)(HLCK)(\mathbf{Vhs/Vliq})]}{(RT)(1.0132\ bar / atm)}$$

Value	Measurement Type	Notes	Reference#
<5 ppb	"DO"	expected value for oxygen-free nitrogen (used for data validation)	4
6.58 ppb	"DO"	this value (achieved at pilot brewery) is "low and repeatable"	5
30 ppb	"DO"	"acceptable value on line" [at undisclosed brewery]	6
30 ppb	"dO2"	"...sub-30 ppb levels in final product are not noticeably detrimental"	10
30 ppb	$dO2_u$	highly achievable value	14
65 ppb	$dO2_u$	"not the sort of level that will cause major oxidation/staling issues"	9
80 ppb	TPO	typical value on bottling line [at undisclosed brewery]	15
100 ppb	$dO2_e$	target maximum value	9
100 oob	TPO	w latest techn it's now possible to produce beers with < 100 ppb TPO	16
110 ppb	TPO	typical value on can line [at undisclosed brewery]	15
<200	TPO	internal UCL for Bell's Brewery packages	14
<250 ppb	$dO2_e$	"maximum in equilibrated, fresh-filled bottle for traditional ale"	7
250 ppb	TPO	glass crushed if greater than this value [at undisclosed brewery]	15
204 ppb	$dO2_u$	"starting to level out" [at this value] (after third day of package testing)	8
250 ppb	TPO	target maximum value; "good TPO is no more than 2.5x dissolved"	9
320 ppb	$dO2_e$	"starting to level out" [at this value] (after third day of package testing)	8
500 ppb	TPO	All brewers should be capable of achieving < 500 ppb in TPO	16
0.25 mL	headspace air	target value during headspace era	14
0.3-0.35 mL	headspace air	average value obtained during headspace era	14
0.5-0.6 mL	headspace air	"for us it is pretty standard" "spec...less than 1 mL air"	11
above 1.0 mL	headspace air	"definitely room for improvement"	12
1.0 mL	headspace air	"anywhere at 1.0 or less is decent. 0.5 or less is great."	13

Table 2. Benchmark values for package oxygen measurements.

$dO2_u$ = DO on an un-shaken package
$dO2_e$ = DO on a shaken package

INSTRUMENTS

-Hach: 3100 ORBISPHERE LDO portable measuring & logging instrument
-Hach: 410 ORBISPHERE controller for M1100 Luminescent Oxygen Sensor
-Hach: 3650 ORBISPHERE Portable ppb EC O2 analyzer
-Hach: 6110 ORBISPHERE ppb EC O2 and CO2 analyzer
-Zahm and Nagel: Series 6000 D.T. Piercing Device
-Steinfurth: Pneumatic sampler

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14. In-house
15. Anonymous

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