Non-destructive optical measurements of dissolved oxygen of filled BIB packages to optimize filling and packaging technology

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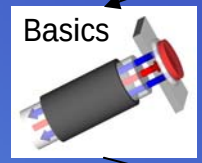
Content

- Who is PreSens?
- Basics of Measurement Technique
- Oxygen Ingress Measurement
- Inline Oxygen Measurement



Who is PreSens

- Founded 1997 as a spin-off of the University of Regensburg
- Market:
  - distributing worldwide chemo-optical sensors and the instrumentation
  - OEM elecronics;
  - contract research;
- 2006: 36 Employees

Biopark Regensburg

Biotech Companies

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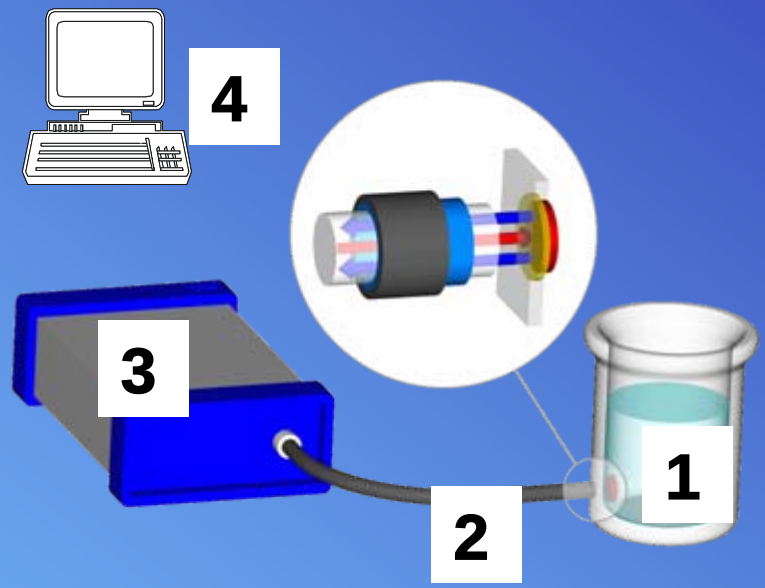
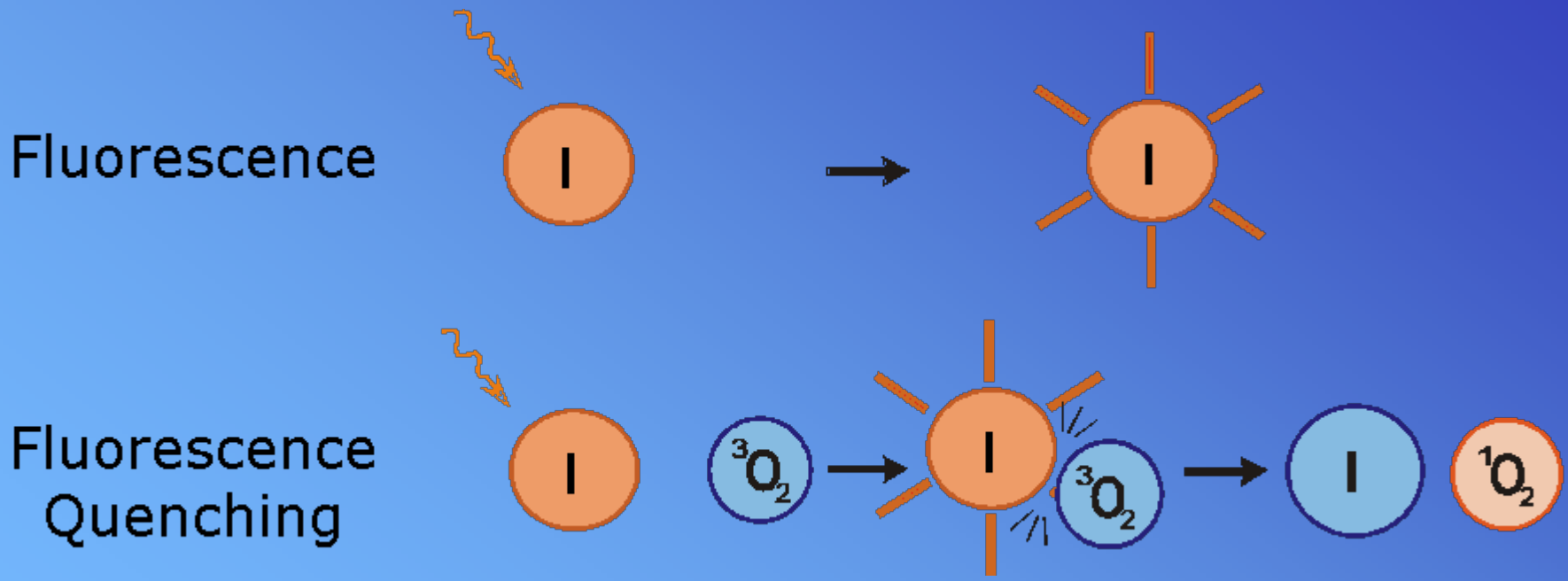
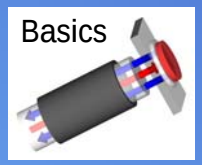
Components to perform non-invasive oxygen measurements



1. Sensor Spot integrated in the vessel /package
2. Optical Fiber
3. Measuring Unit
4. Computer

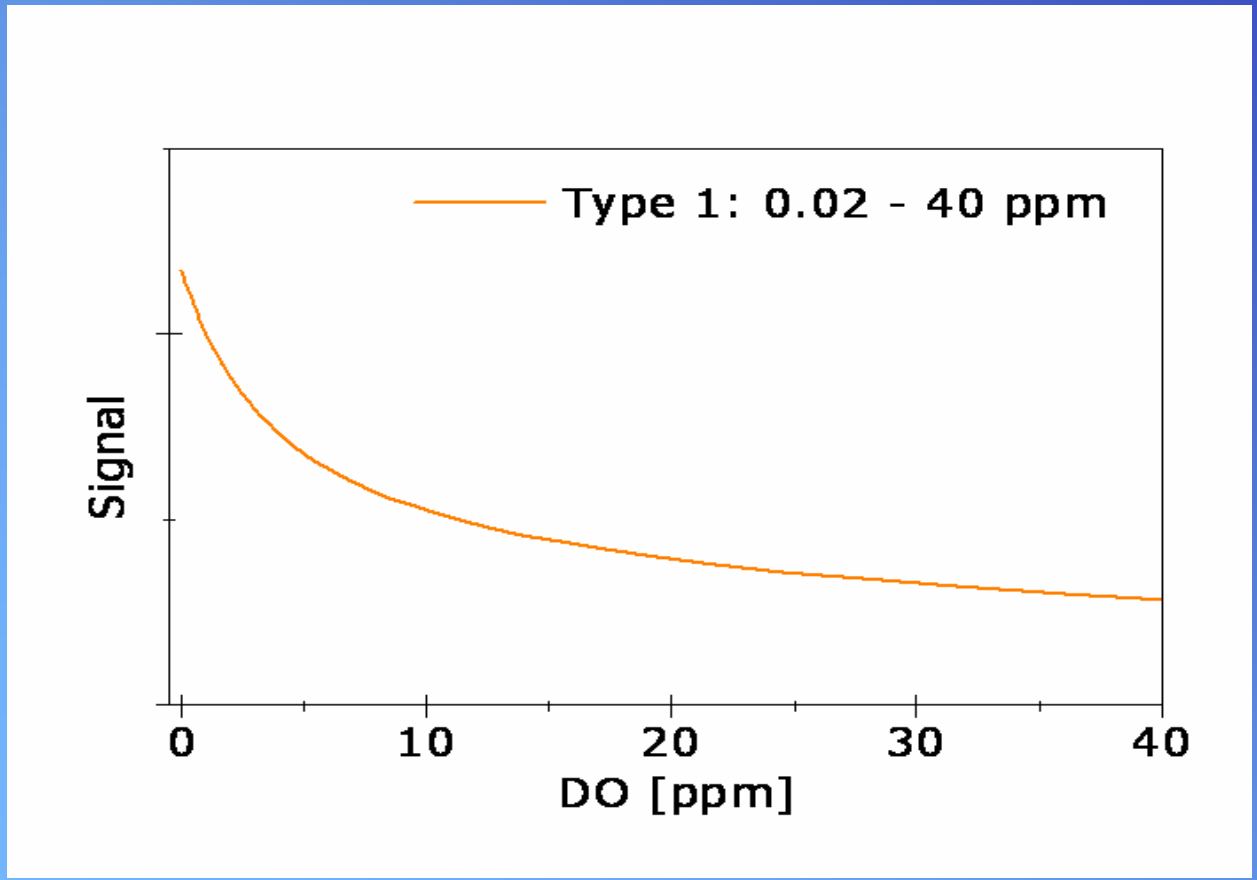
Dynamic fluorescence quenching

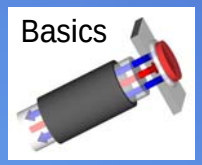




Measurement principle

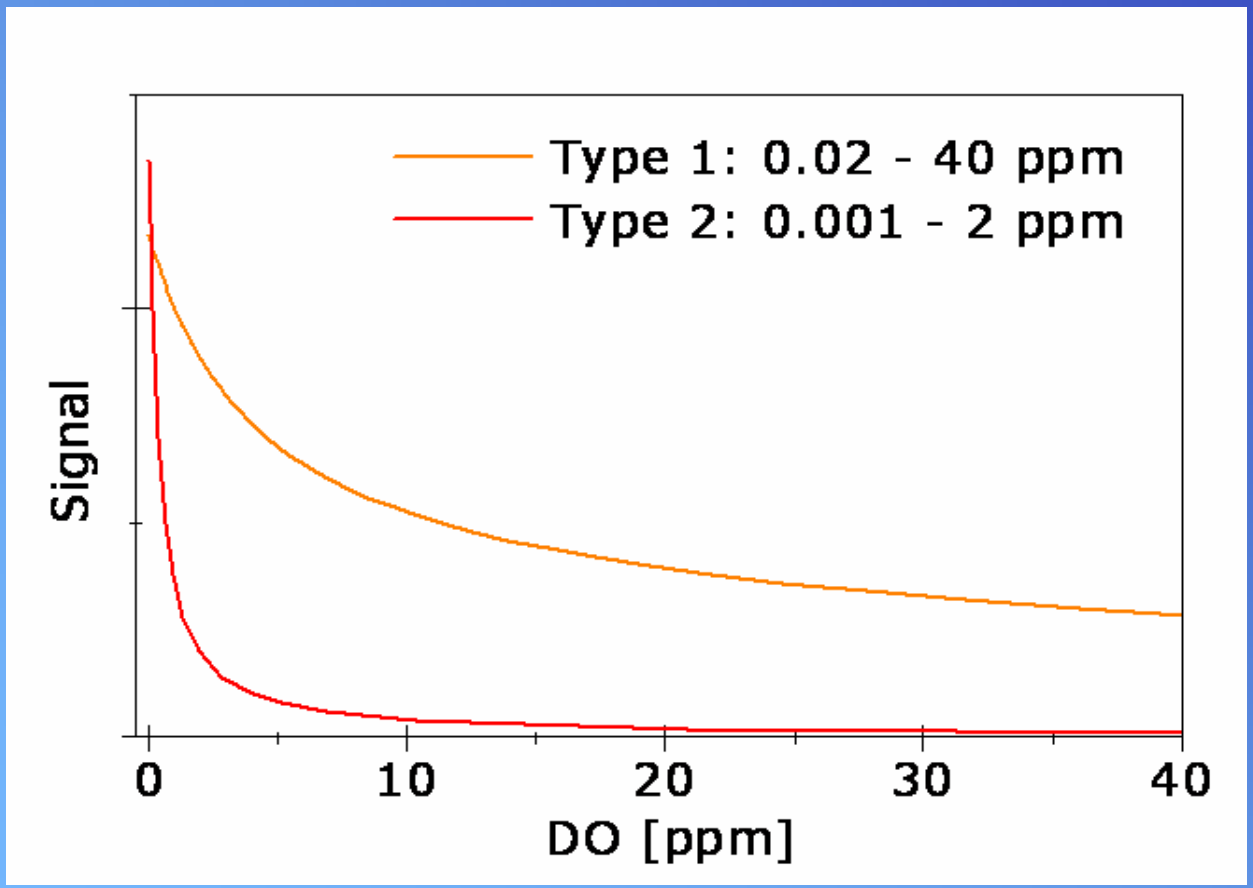
Type 1: Oxygen sensor





Measurement principle

Type 1: Oxygen sensor
Type 2: Trace oxygen sensor



1. Oxygen Ingress Measurment into plastic containers
   - non-invasive
   - non-destructive
   - measurement under real conditions

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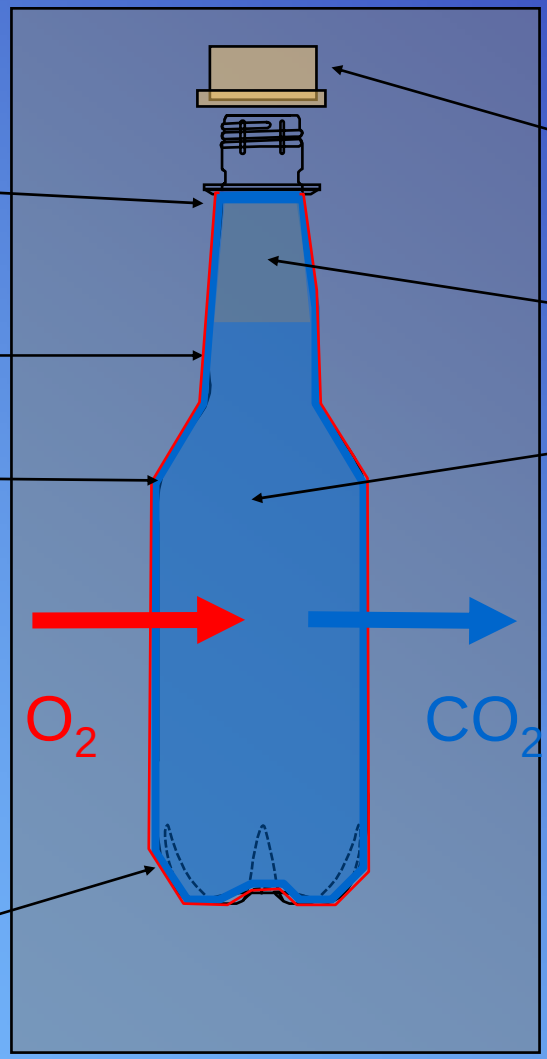
PET bottles – shelf life factors

CONTAINER

- PET resin (Scavenger)
- Wall thickness
- Preform weight
- Surface/volume ratio of bottle
- Blowing process
- Blow molds
- Coating (internal/external)



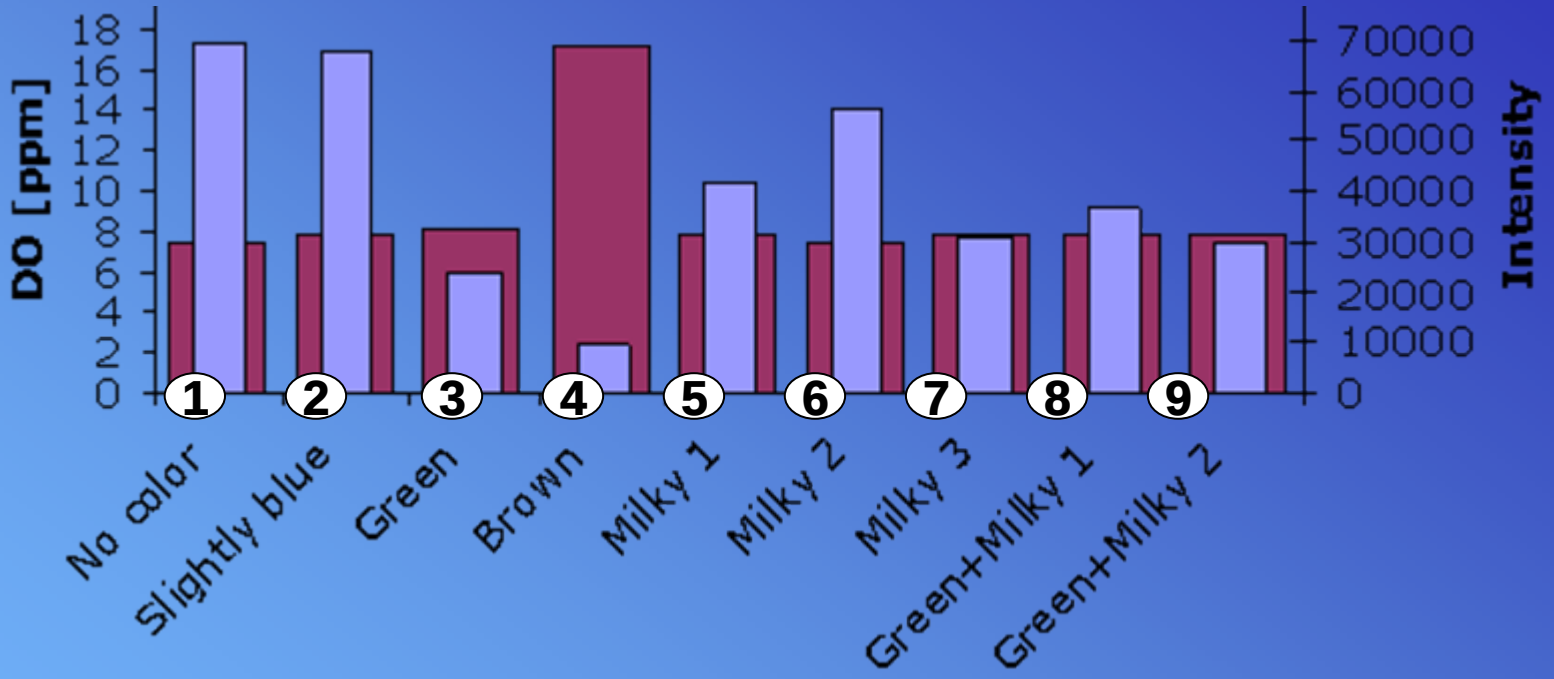
FILLING

- Closure material (Scavenger)
- Head space
- Product
- Filling process (N2-droplet, Foaming)
- Pressurization

ENVIRONMENT

- Light
- Storage temperature
- Storage humidity

Measurement with different PET bottles



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Measurement with different PET bottles

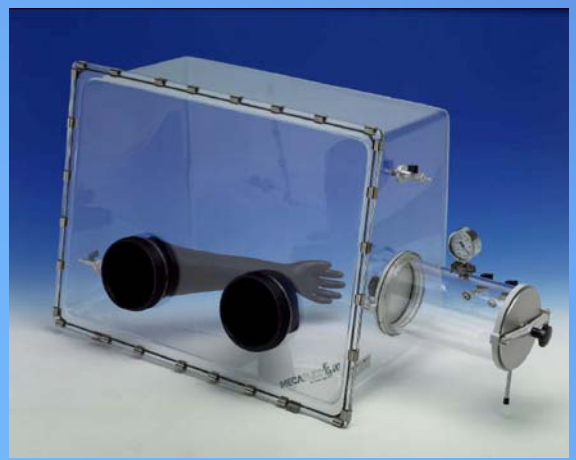
+

Integration of a transparent closure containing the sensor spot to measure in strongly colored or non-transparent containments

System components

Measuring unit (1, 4, 10 channel)

Fiber, closure mount



Option 1: Glove box

Option 2: Dosing pump

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Ensure equilibrium

Shaker control of temperature

Environmental chamber

control of temperature, RH

Definitions

OTR (Oxygen Transmission Rate) :

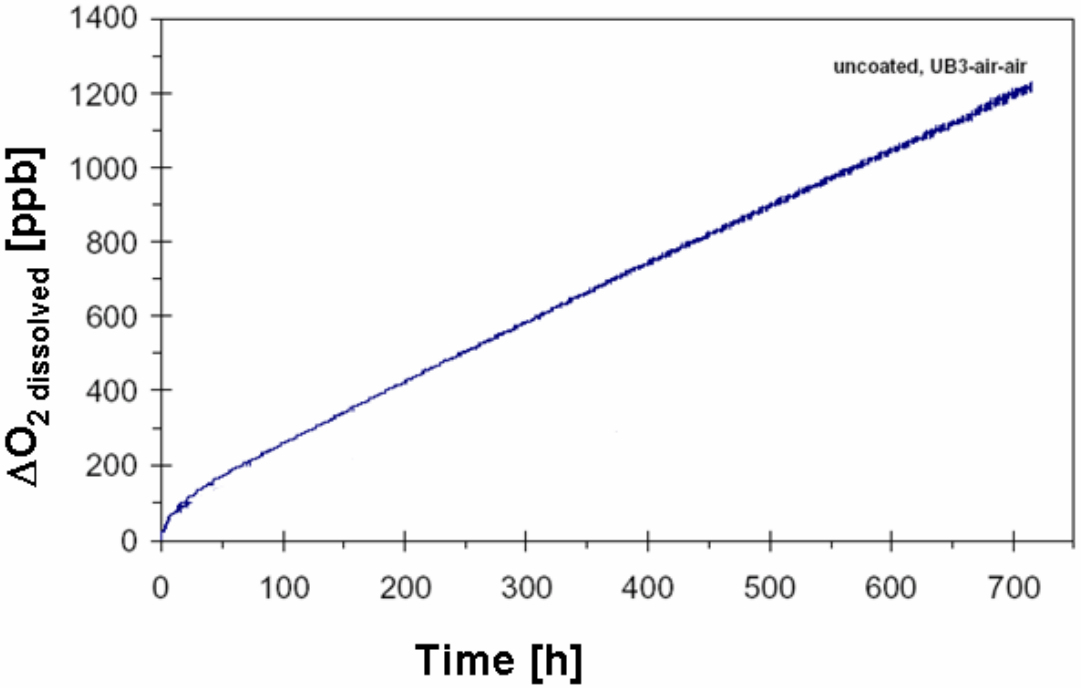
partial O_2 ingress per time ($\Delta P / \Delta t$)

BIF (Barrier Improvement Factor) :

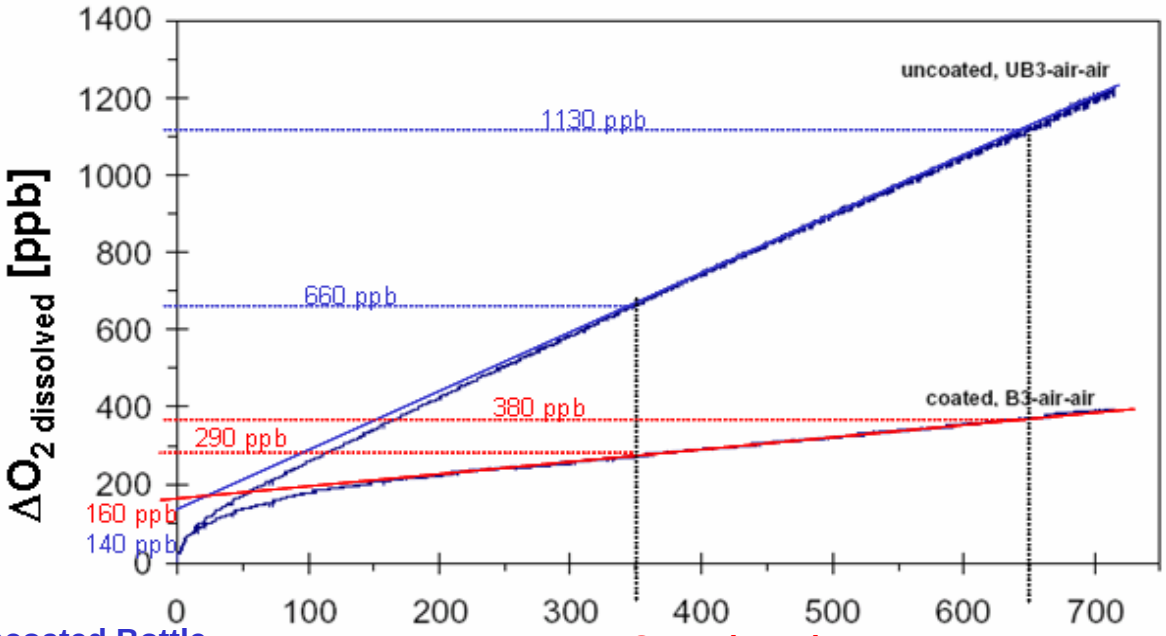
$$BIF(O_2) = \frac{\text{OTR of uncoated bottle}}{\text{OTR of coated bottle}}$$

Shelf life : time to reach O_2-ingress or CO_2 -loss limits in bottle contents

Outer Coatings



Outer Coatings



Uncoated Bottle

$O2(t)_{diss}$=140 ppb+37,60 ppb/d*t

Time for 1ppm total O2 ingress: 7.4 days

Coated Bottle

$O2(t)_{diss}$ = 160 ppb + 7,20 ppb/d * t

Time for 1ppm total O2 ingress :35.6 days

BIF(O_2) = 37,60 / 7,20 = **5,2**

Measurement parameter

Bottle volume, head space (HS)	500ml, 20 ml
Temperature	30 °C
Relative humidity	50 %
Bottle contents	N_2-Water
CO_2 contents	0 g/L (no carbonation)
Bottle weight	38 g
Scavenger contents	0 %

Conlusion :

- Limit of total O2 ingress : 1000 ppb in 180 days !
- Coating without scavenger : the total O2-limit is already reached after 36 days -> scavenger is necessary !!

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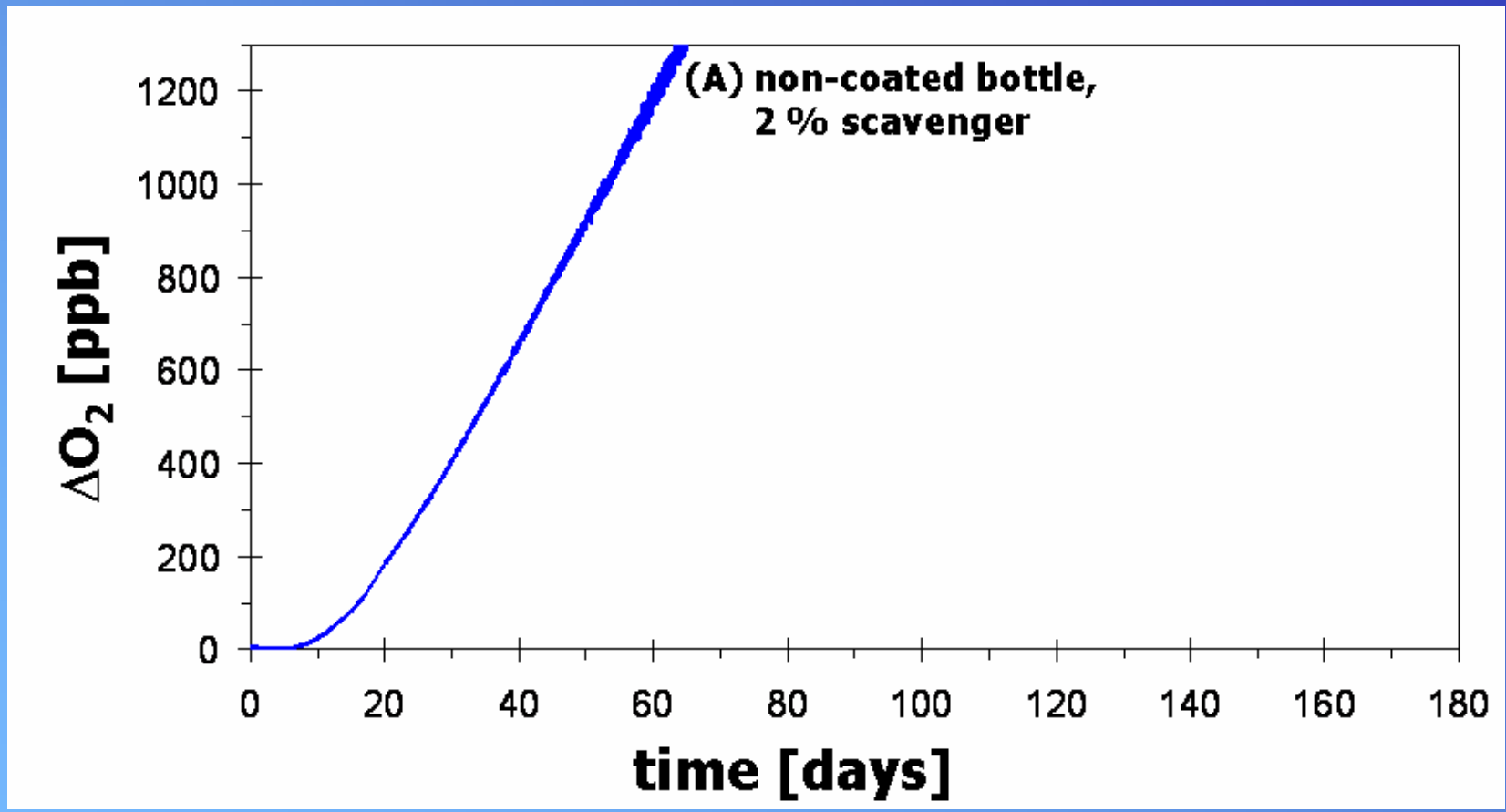
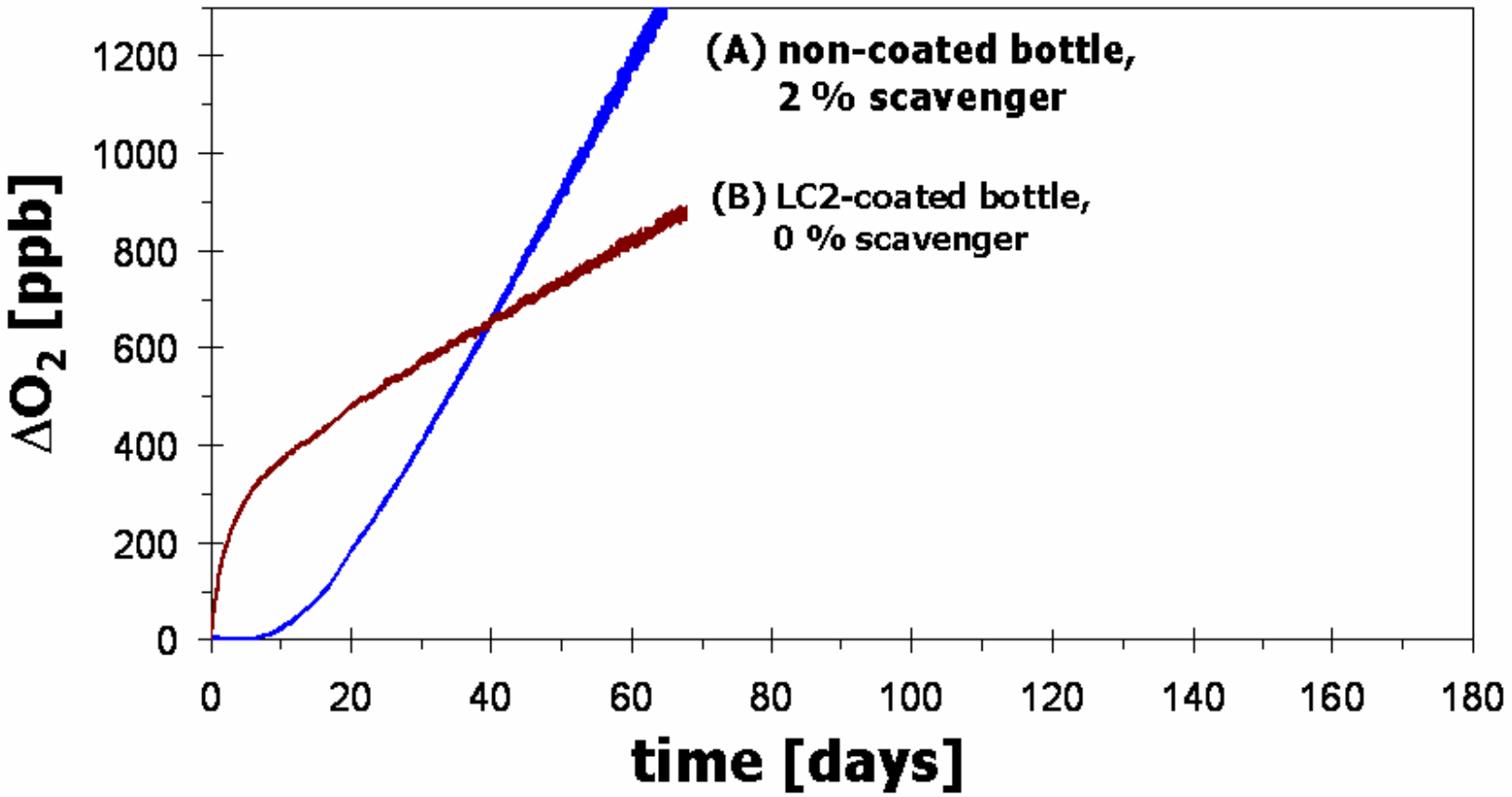
Comparison of different treated PET bottles:
(A) only scavenger



Comparison of different treated PET bottles:
(A) *only scavenger* – (B) only outer coating



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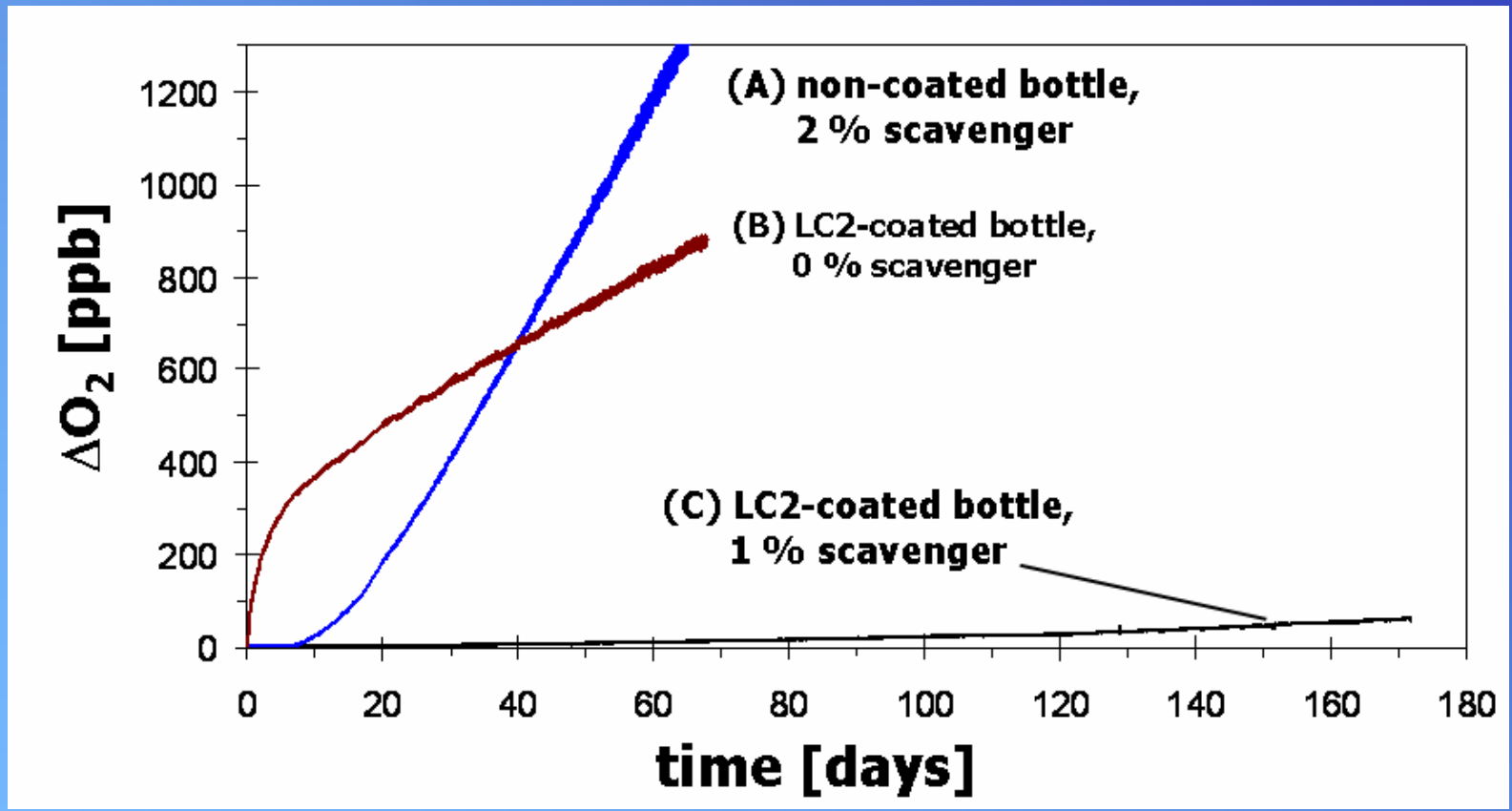
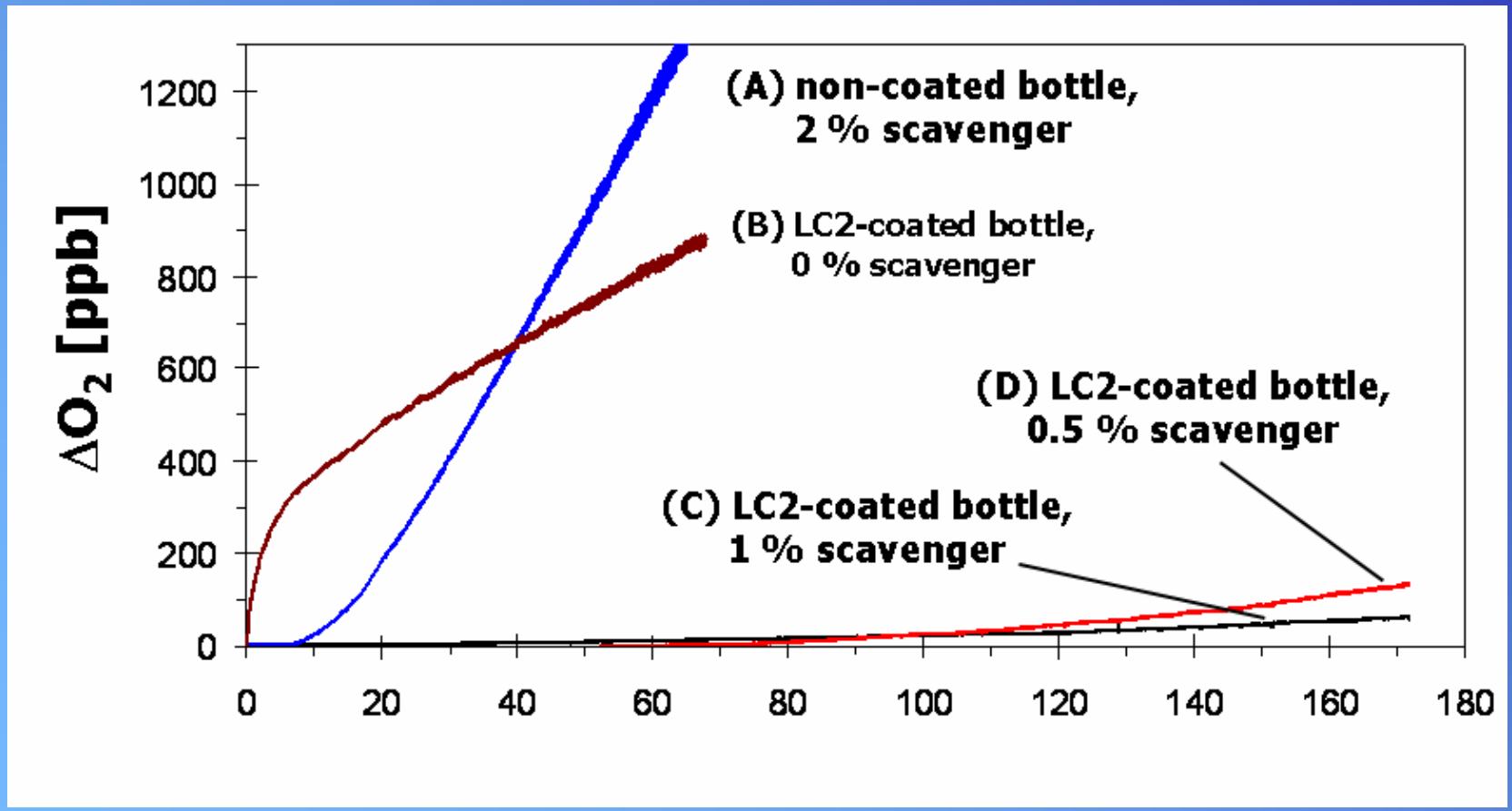
Comparison of different treated PET bottles:

(A) *only scavenger* – (B) only outer coating – (C) scavenger + outer coating (1 %)

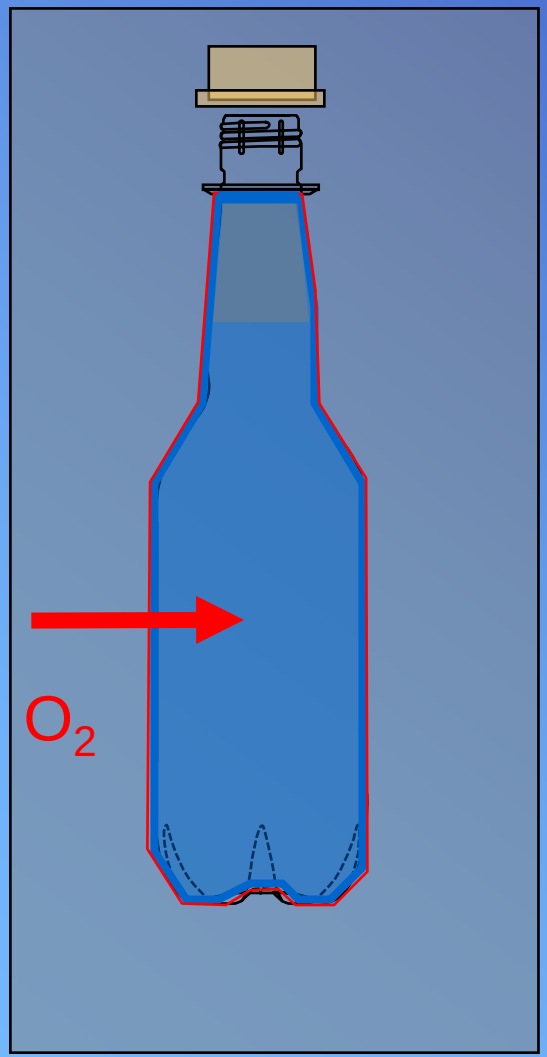


Comparison of different treated PET bottles:

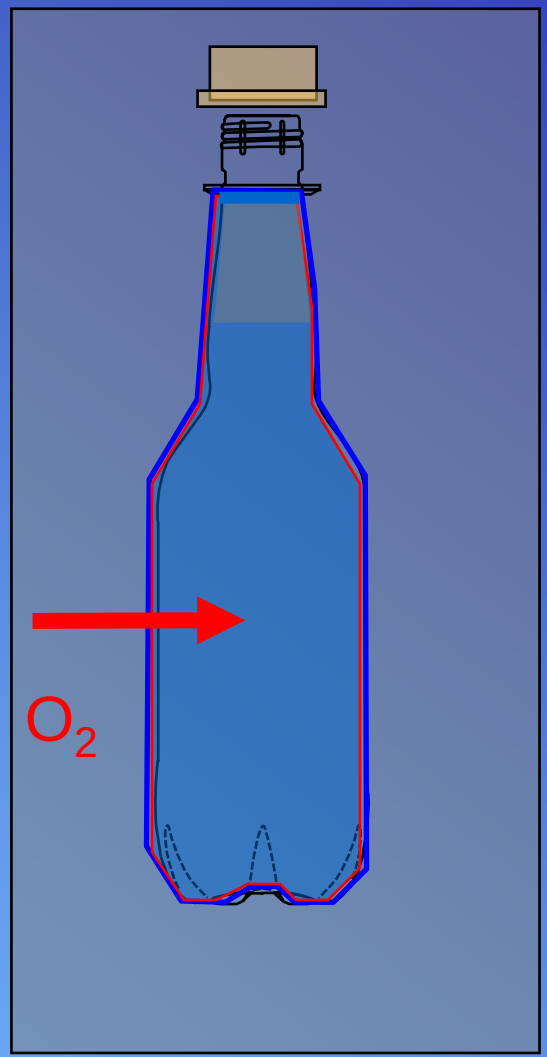
(A) *only scavenger* – (B) only outer coating – (C,D) scavenger + outer coating (1%, 0.5%)



Outer Coating



Inner Coating



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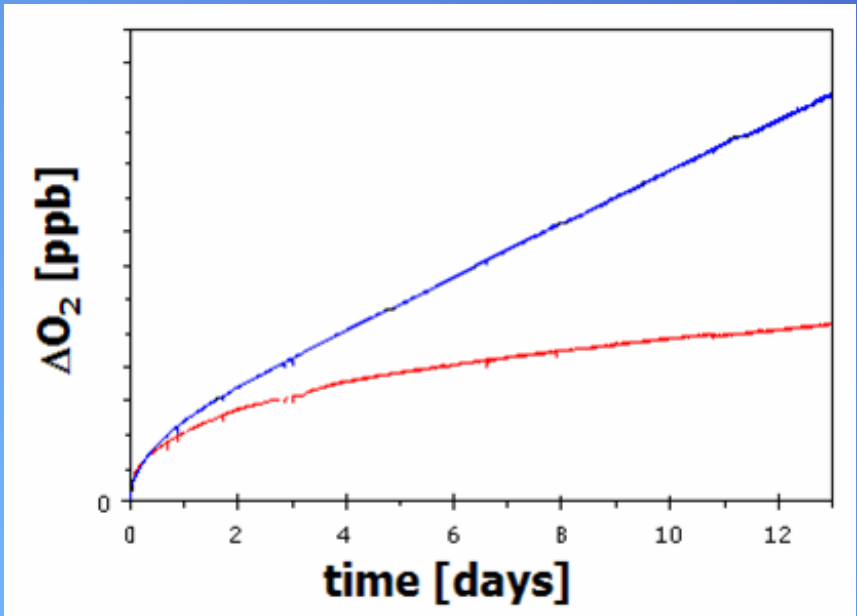
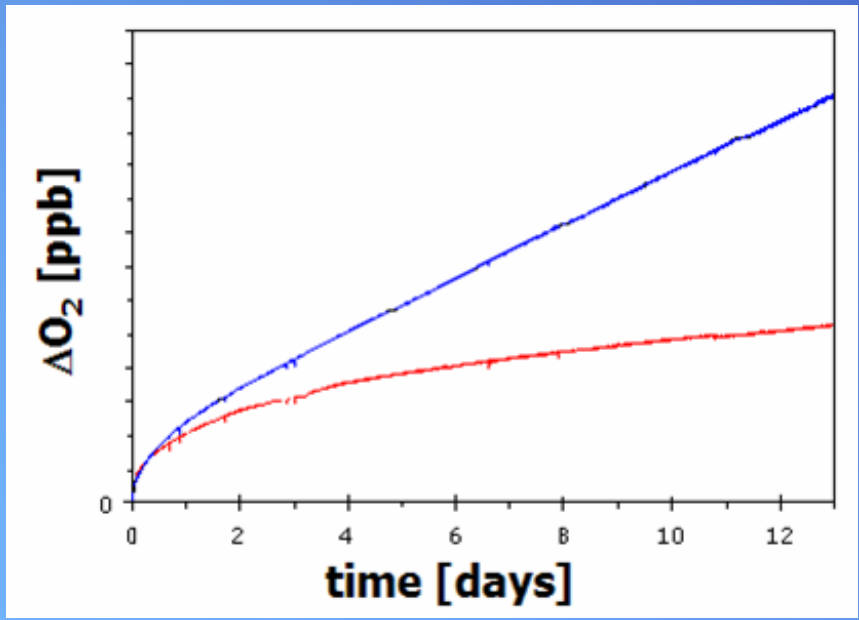
Outer Coating



- No contact with content
- Recoating possible

Outer Coating



- No contact with content
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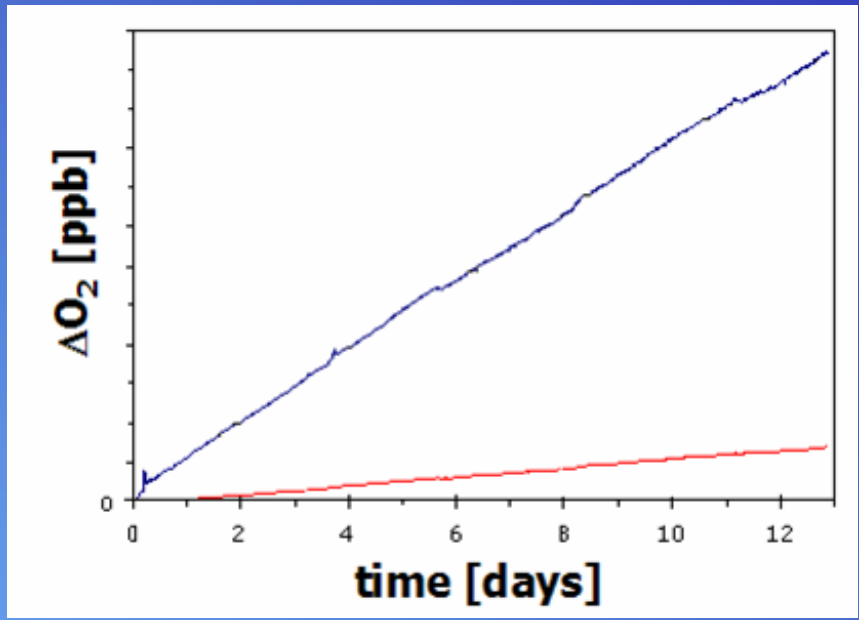
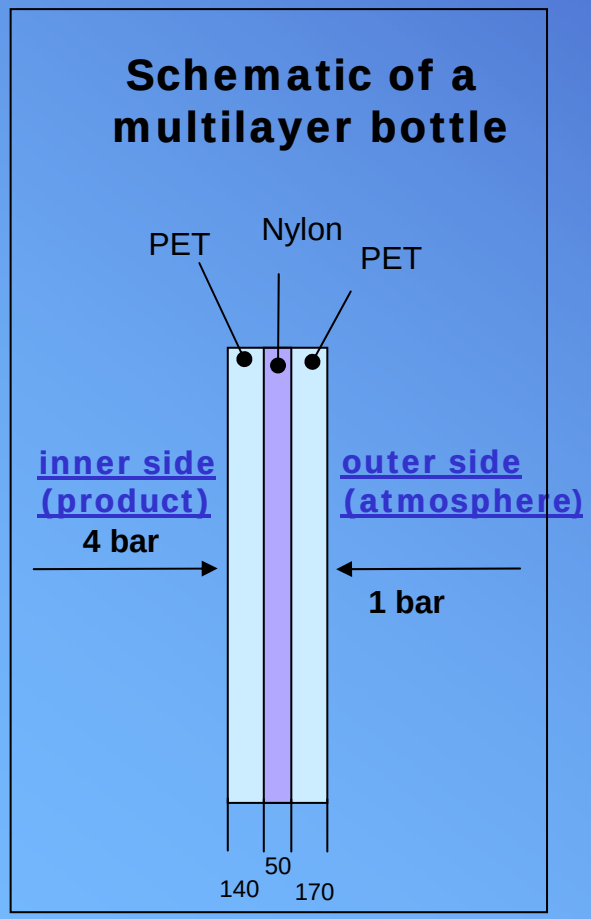
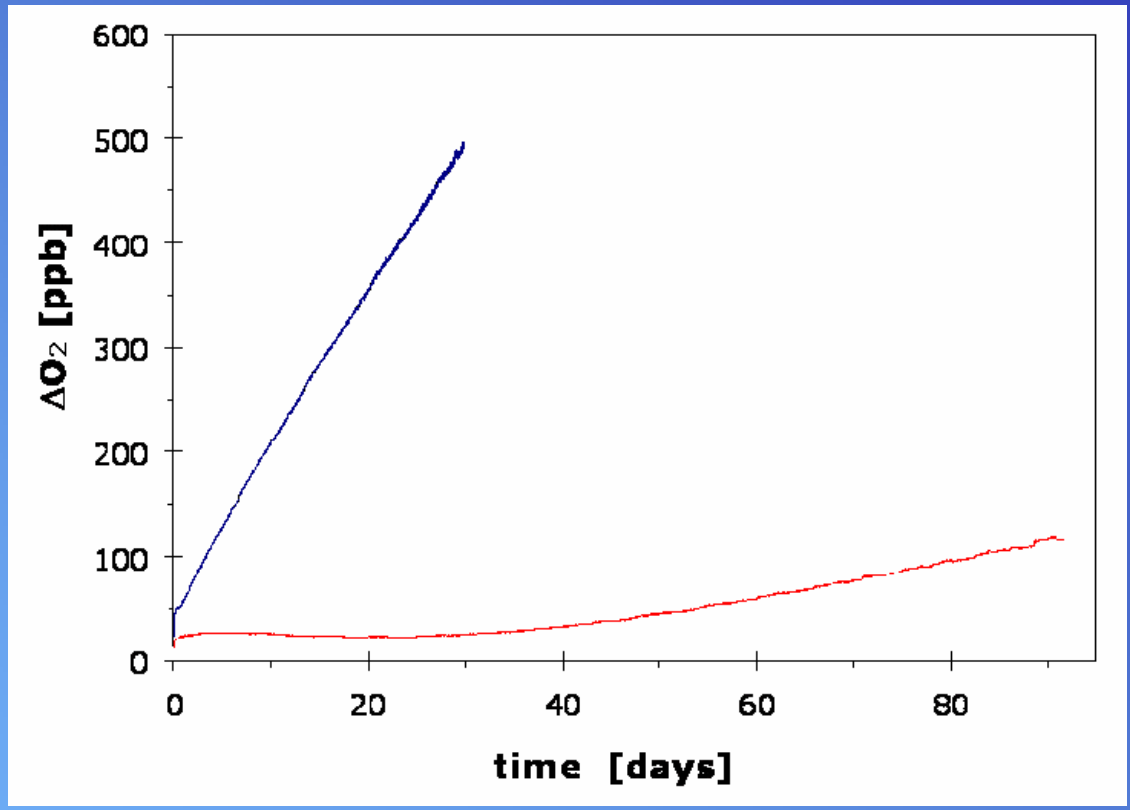
Inner Coating



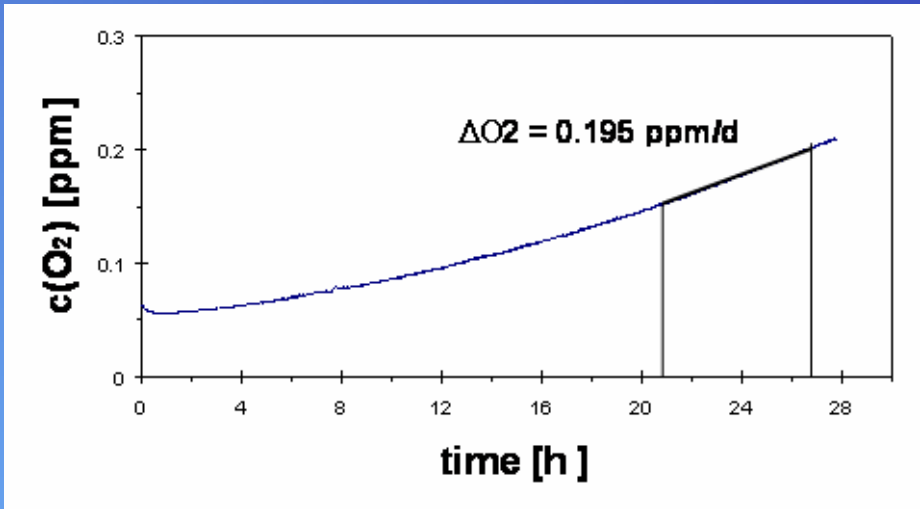
- Reduced oxygen ingress from bottle reservoir

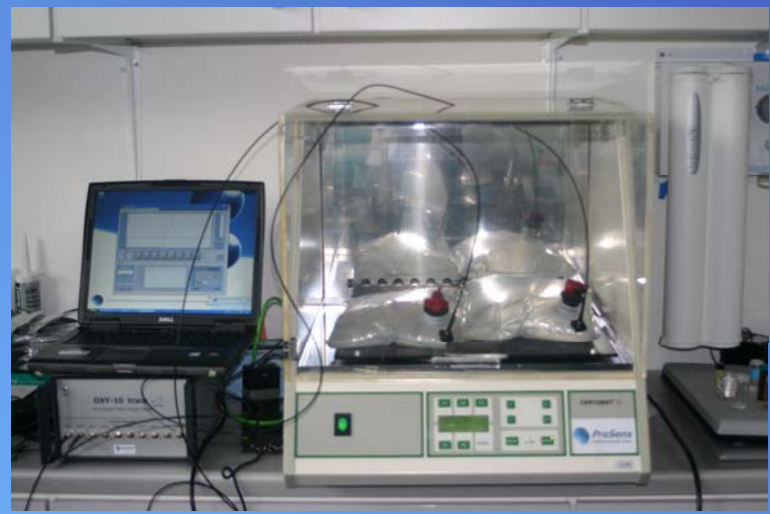
Multilayer Bottles





Oxygen Ingress Measurment in the Bag-In-Box System





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2. Measurement of oxygen in real probes (beer, wine)

Measurement in the brewing process

1) DO in bottles with swing stoppers

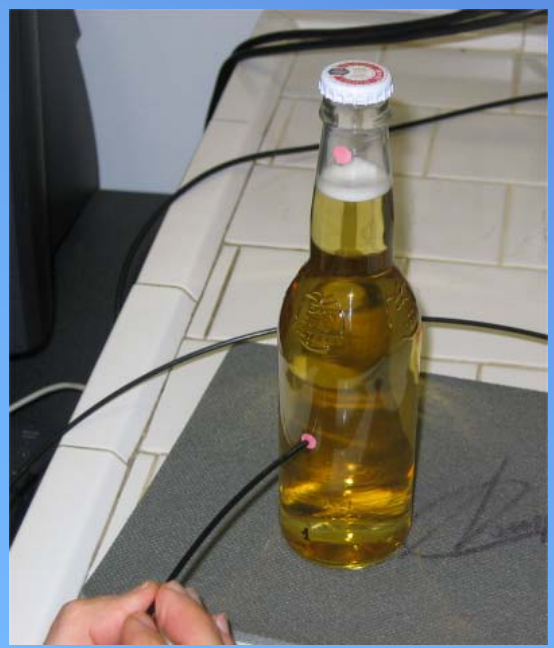
Not possible: Comparison with (standard) electrode

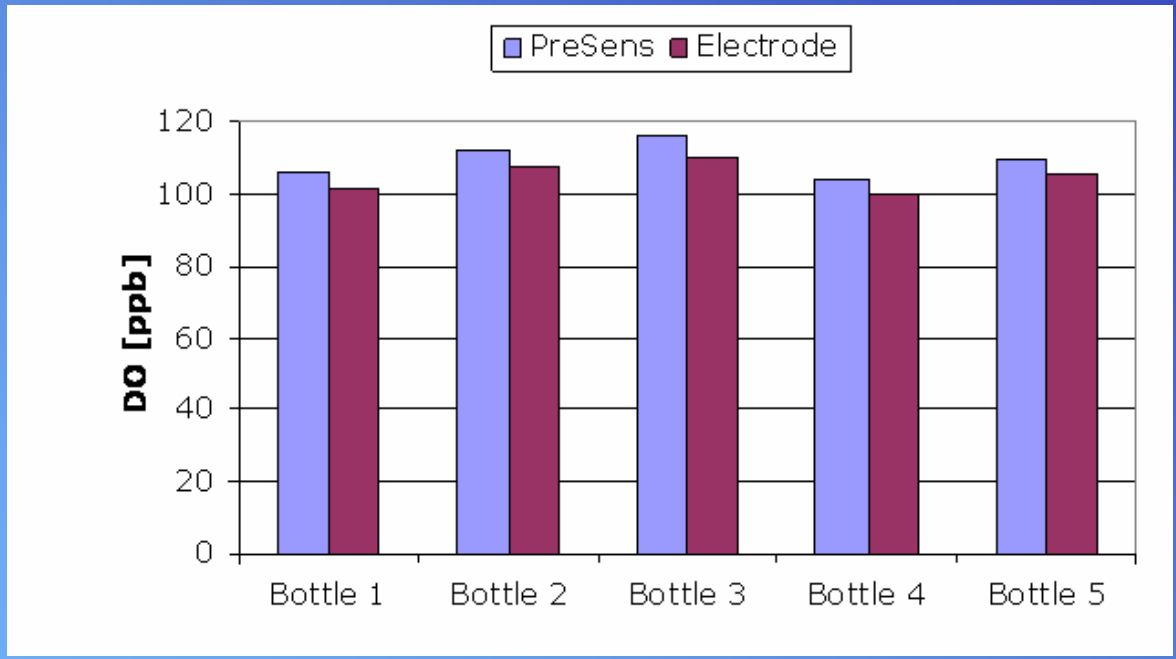
→ [DO] significantly higher than in standard bottles

Measurement in the brewing process

2. DO in bottles

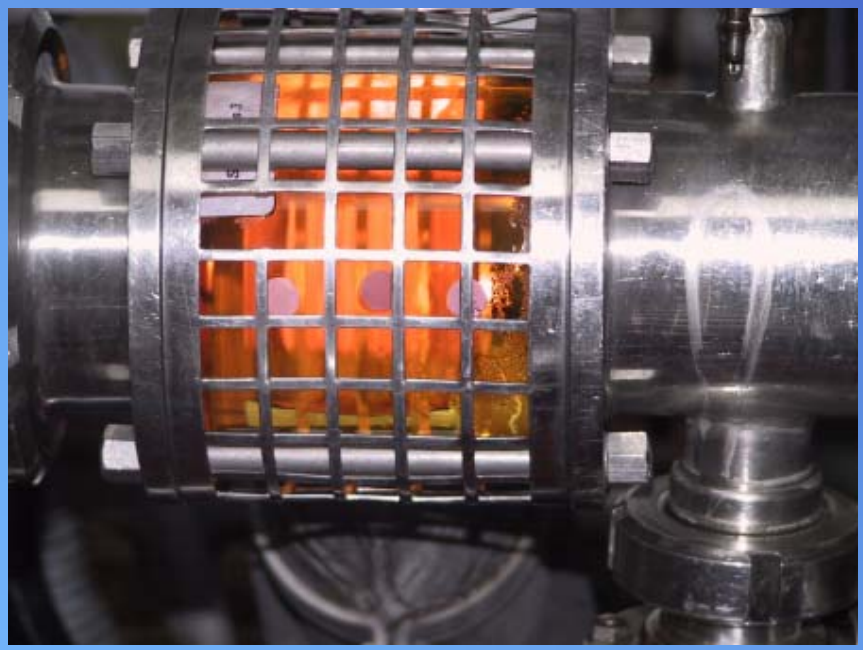
Comparison with (standard) electrode





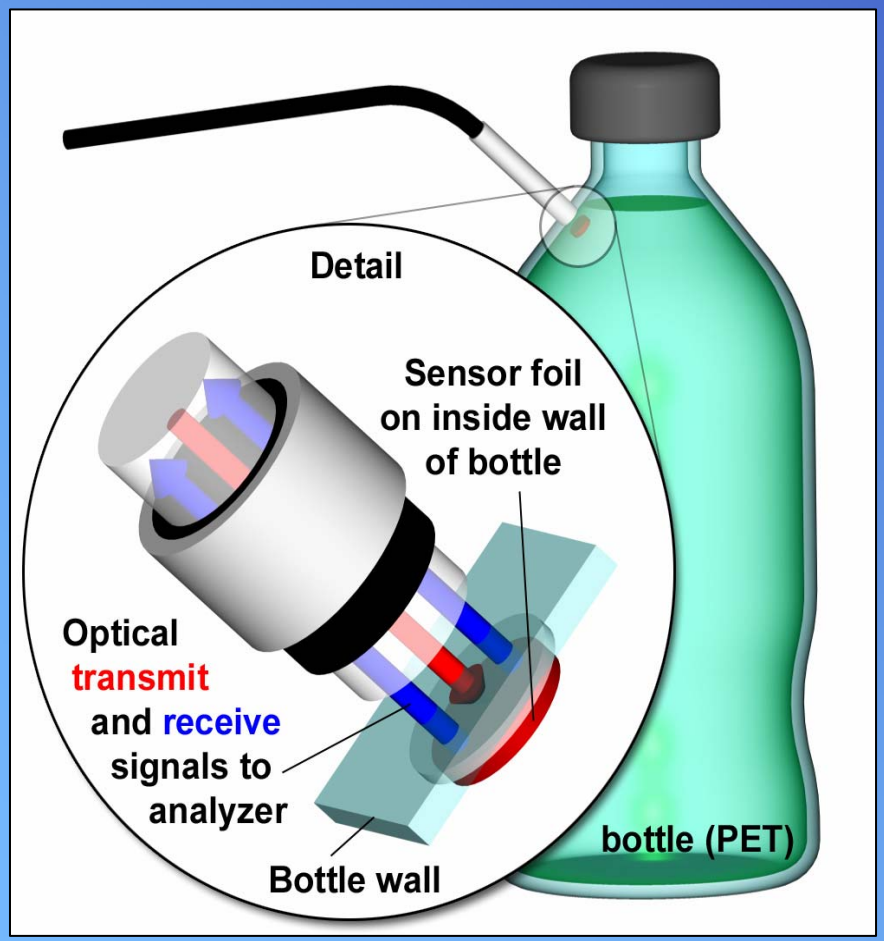
Measurement in the brewing process

3) DO measurement through glass



- Sensor patches attached to the inner surface of inspections glasses
- Simple and fast measurement by placing the fiber to the spot
- Fast possibility to measure the actual oxygen concentrations at different locations

Conclusions - PET



- Non-invasive
- Non-destructive
- High precision
- Detection limit 1ppb dissolved oxygen
- Liquid or gas phase (headspace) measurement
- Online measurement, trend analysis
- Robust, long term stability (more than 100.000 data point without drift)
- Exclude closure influence
- Real life measurement conditions (Temperature, Humidity, Pressure, Contents)

Conclusions – Ínline Measurement

- Stands CIP & SIP
- High shelf of membrane 6 months
- No need for recalibration
- No cross-sensitivity to CO2
- Detection limit 1ppb dissolved oxygen
- Liquid or gas phase (headspace) measurement

Thank you for your attention

Bon appétit.