

### Annual meeting June 14-17, 2015 La Quinta, California

## The Evolution of CO<sub>2</sub> Measurement

# What does it bring breweries and what is the next step?

Frank Verkoelen, Pentair Haffmans Week 25 -2015

#### Content:

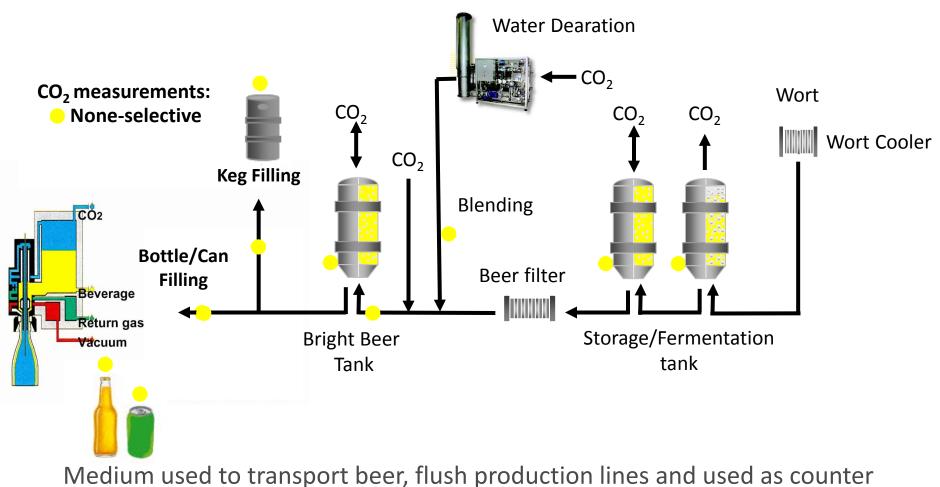
- Applications
- Setting the standard
- CO<sub>2</sub> measurement, General
- Influence factors
- CO<sub>2</sub> measurement of packages
- New technologies
- Calibration/Verfication
- Good practice

## How does a brewery determine the optimum gas content for a new product development?



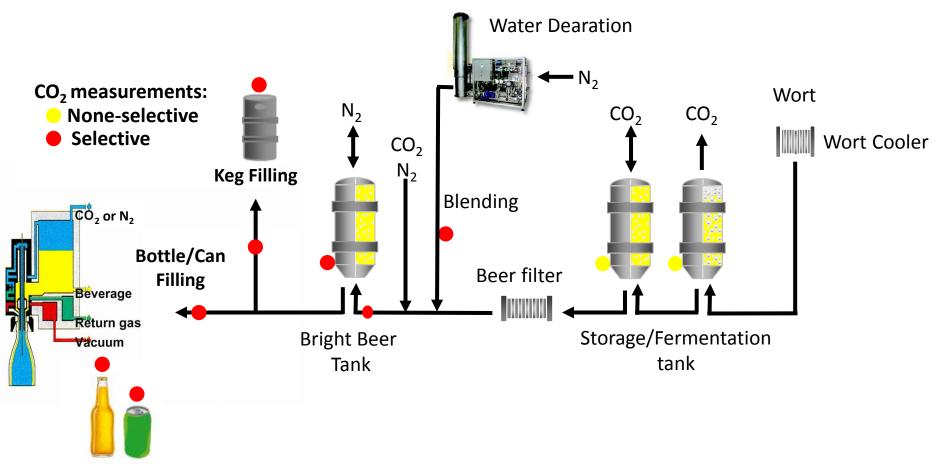
#### By Tasting (Tasting Panels)!

### CO<sub>2</sub> MEASUREMENT, Typical Applications with CO<sub>2</sub>



pressure on tanks is important in the choice of the CO<sub>2</sub> measurement.

### CO<sub>2</sub> MEASUREMENT, Typical Applications with N<sub>2</sub>



Medium used to transport beer, flush production lines and used as counter pressure on tanks is important in the choice of the CO<sub>2</sub> measurement.

The Science of Beer

The Evolution of CO<sub>2</sub> Measurement

#### CO<sub>2</sub> MEASUREMENT



### CO<sub>2</sub> METHODOLOGY

Which standard CO<sub>2</sub> Measuring Methodology is followed?

### ASBC Method of Analysis or Analytica-EBC?

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CO2 Measurement & Methodology

### Evolution of CO<sub>2</sub> MEASUREMENT

#### Evolution of CO<sub>2</sub> MEASUREMENT results in a wide range of CO<sub>2</sub> Measuring Methods

CO <sub>2</sub> measurement methods	ASBC Methods	EBC Methods
- Manometric (mechanical)		
- P&T	beer-13	9.28.3
- Multi-volume expansion	beer-13	9.28.5/6
- Membrane		
- Manometric (optical)		
- Adsorption		
- None invasive		
- Chemical/titrimetric		
- Blom & Lund		9.28.1
- Titration (Corning)		
- Special CO <sub>2</sub> detectors		
- Thermal conductivity		9.28.4
- Attenuated Total Refrection ATR		

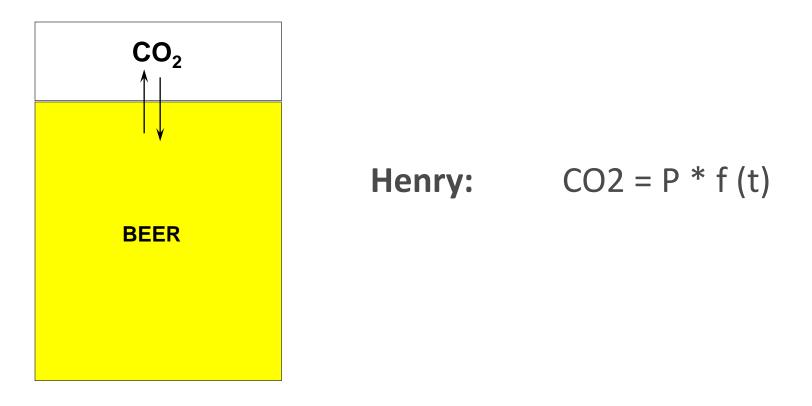
### CO<sub>2</sub> MEASUREMENT

Estimated 90 % of CO<sub>2</sub> MEASUREMENT currently used in brewing industry:

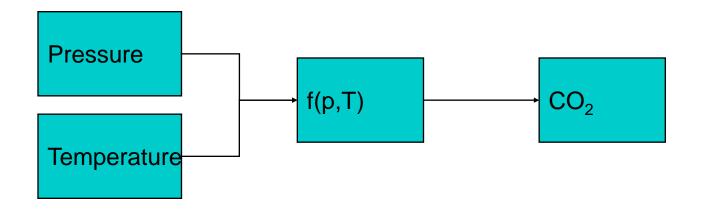
### Henry's Law

"The volume of gas dissolved in a liquid is proportional to the pressure of the gas above the liquid "

#### CO2 MEASUREMENT BASED ON HENRY'S LAW



#### FUNCTIONAL DIAGRAM HENRISCH LAW



GAGE PRESSURE – POUNDS PER SQUARE INCH	ASBC CO <sub>2</sub> CHART		
"F 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24			
32 2.15 2.27 2.38 2.48 2.59 2.70 2.88 2.90 3.00 3.11 3.21	SOLUBILITY OF CARBON DIOXIDE IN BEER		
33 2,10 2,23 2,33 2,43 2,53 2,63 2,74 2,84 2,96 3,06 3,15 3,25	PRESSURE—TEMPERATURE RELATIONSHIPS		
34         2.06         2.18         2.28         2.38         2.48         2.58         2.69         2.79         2.90         3.00         3.69         3.19	Results Expressed as Volumes CO <sub>2</sub>		
35 2.02 2.14 2.24 2.34 2.43 2.52 2.63 2.73 2.83 2.93 3.02 3.12 3.22	(°C—760 mm) per Volume Beer		
36         1.98         2.09         2.19         2.38         2.47         2.57         2.67         2.77         2.86         2.96         3.65         3.45         3.24           37         1.94         2.04         2.24         2.33         2.42         2.52         2.62         2.71         2.80         3.69         3.09         3.18         3.27	( C=700 mm) per volume beer		
37         1.94         2.04         2.14         2.23         2.42         2.52         2.62         2.71         2.80         2.90         3.09         3.18         3.27           38         1.90         2.00         2.10         2.29         2.33         2.42         2.57         2.66         2.75         2.85         2.94         3.03         3.12         3.21			
30         1.56         2.46         2.15         2.25         2.34         2.43         2.52         2.61         2.70         2.88         2.89         2.98         3.07         3.16         3.25         3.25	CONVERSION SCALE		
<b>40</b> 1.83 1.92 2.01 2.10 2.20 2.30 2.47 2.56 2.65 2.75 2.84 2.93 3.01 3.10 3.19 3.28	(sp gr beer, 1.01)		
41 1.79 1.88 1.97 2.06 2.16 2.25 2.34 2.43 2.52 2.60 2.79 2.79 2.88 2.96 3.05 3.14 3.23	· · · · · · · · · · · · · · · · · · ·		
42 1.75 1.85 1.94 2.02 2.12 2.21 2.30 2.39 2.48 2.56 2.65 2.74 2.83 2.91 3.00 3.09 3.18 3.26	· · · · · · · · · · · · · · · · · · ·		
43 1.72 1.81 1.90 1.99 2.08 2.17 2.26 2.34 2.43 2.52 2.61 2.69 2.78 2.86 2.96 3.84 3.13 3.21			
	25 26 27 28 29		
45 1.66 1.75 1.84 1.91 2.00 2.08 2.17 2.26 2.34 2.42 2.51 2.60 2.69 2.77 2.86 2.94 3.02 3.11 3.19			
46         1.62         1.71         1.88         1.96         2.14         2.13         2.22         2.38         2.47         2.55         2.64         2.72         2.81         2.89         2.98         3.06         3.15         3.23			
47 1.59 1.68 1.76 1.84 1.92 2.00 2.09 2.18 2.26 2.34 2.42 2.50 2.59 2.67 2.76 2.84 2.93 3.02 3.09 3.18			
48         1.56         1.65         1.73         1.81         1.89         1.96         2.05         2.14         2.22         2.30         2.38         2.46         2.54         2.62         2.71         2.79         2.88         2.96         3.04         3.13           49         1.53         1.62         1.70         1.79         1.86         1.93         2.10         2.18         2.25         2.34         2.42         2.58         2.67         2.75         2.33         2.91         3.00         3.07         2.30         3.04         2.42         2.59         2.58         2.67         2.75         2.33         2.91         3.00         3.07         3.00         3.07         3.00         3.07         3.00         3.07         3.00         3.07         3.00         3.07         3.00         3.00         3.07         3.00         3.07         3.00         3.07         3.00         3.07         3.00         3.07         3.00         3.00         3.07         3.00         3.00         3.07         3.00         3.00         3.07         3.00         3.00         3.00         3.00         3.00         3.00         3.00         3.00         3.00         3.00         3.00 </td <td>3.15 30 31 32 33 34</td>	3.15 30 31 32 33 34		
	3.15 30 31 32 33 34 3.10 3.17		
	3.06 3.13 3.19		
	309 J.06 J.13 J.22		
	2.94 3.01 3.09 3.16		
	2.89 2.96 3.04 3.10 3.17 35 36 37 38 39		
	2.83 2.89 2.97 3.04 3.11 3.18		
56         1.50         1.57         1.65         1.72         1.79         1.86         1.93         2.00         2.08         2.15         2.22         2.29         2.36         2.43         2.50         2.57         2.64         2.71         2.64           57         1.54         1.62         1.77         1.83         1.90         1.97         2.04         2.11         2.18         2.25         2.32         2.39         2.46         2.53         2.60         2.66         2.71 </td <td>2.78 2.35 2.92 2.99 3.66 3.13 3.20</td>	2.78 2.35 2.92 2.99 3.66 3.13 3.20		
	2.73 2.80 2.87 2.94 3.00 3.08 3.15 3.22		
▌ <b>▐</b> <mark><mark><mark>╴</mark>╋<del>╴╡╸╡╸╡╸╡╸╡╸╡╸╡</del>╶╋╴╴╋╴╴╋╴╴╋╴╴╋╴╴╋╴╴╋╴╴╋╴╴╋╴╴╋╴╴╋╴╴╋╴╴╋╴╴</mark></mark>	2.69 2.75 2.82 2.88 2.96 3.02 3.09 3.16		
	2.64 2.70 2.77 2.84 2.91 2.97 3.03 3.10 3.17		
60         1.54         1.62         1.69         2.75         1.82         1.88         1.95         2.01         2.08         2.14         2.21         2.27         2.34         2.40         2.47         2.53         1           61         1.51         1.59         1.66         1.72         1.79         1.85         1.91         1.97         2.04         2.10         2.17         2.34         2.40         2.47         2.53         1			
b1         L32         L33         L30         L32         L33         L33 <thl33< th="">         L33         <thl33< th=""> <thl33< th=""> <thl33< th=""></thl33<></thl33<></thl33<></thl33<>	2.56 2.62 2.69 2.75 2.81 2.87 2.94 3.00 3.07 3.14 3.19		
63         1.54         1.60         1.66         1.72         1.78         1.85         1.91         1.98         2.04         2.16         2.22         2.28         2.35         2.41         2	2.48 2.53 2.60 2.66 2.72 2.78 2.85 2.90 2.97 3.03 3.89 3.16		
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65 1.55 1.61 1.67 1.73 1.79 1.85 1.91 1.96 2.03 2.49 2.15 2.21 2.27 2.33 2	2.39 2.45 2.57 2.63 2.69 2.75 2.81 2.87 2.93 3.00 3.06 3.71 3.18		
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67 1.58 1.55 1.61 1.67 1.73 1.79 1.85 1.90 1.96 2.82 2.05 2.14 2.20 2.25 2	2.31 2.37 2.43 2.49 2.55 2.61 2.67 2.72 2.78 2.84 2.90 2.96 3.02 3.07 3.13 3.19		
68         1.53         1.59         1.64         1.70         1.76         1.83         1.87         1.93         1.98         12.44         2.10         2.22         2			
69 1.5J 1.57 1.62 1.68 1.73 1.79 1.84 1.90 1.96 2.0J 2.07 2.13 2.18 2			
	2.21 2.26 2.31 2.36 2.42 2.48 2.54 2.59 2.65 2.70 2.76 2.81 2.87 2.92 2.98 3.03 3.09 3.14		
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	2.14         2.19         2.24         2.25         2.46         2.46         2.57         2.62         2.67         2.72         2.78         2.83         2.89         2.94         3.00         3.05         3.10         3.15           2.17         2.16         2.21         2.26         2.32         2.36         2.42         2.47         2.53         2.68         2.63         2.68         2.79         2.84         2.89         2.94         3.00         3.06         3.10         3.15		
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	1.70 1.80 1.83 1.89 1.94 0.98 2.00 2.07 2.12 2.16 0.21 2.25 2.29 2.33 2.38 2.43 2.47 2.51 2.56 2.68 2.65 2.69 2.74 2.73 2.82 2.87 1.74 1.78 1.82 1.87 1.91 1.95 2.60 2.64 2.69 2.94 2.99 2.93 2.94 2.96 2.99 2.44 2.46 2.63 2.67 0.61 2.46 2.40 2.73 2.78 2.89		

#### 00 CHADT

CALCULATION OF CO<sub>2</sub> EQUILIBRIUM CONCENTRATION according to **ASBC** 

# $\begin{aligned} \text{CO}_2 \left[ \text{Vol} \right] = & 3.45778 - 7872.43^{\circ}\text{P}^{\circ}10^{-5} + 1513.53^{\circ}\text{T}^{\circ}10^{-4} + 7779.99^{\circ}\text{P}^{2*}10^{-7} + \\ & 3963.09^{\circ}\text{T}^{2*}10^{-7} - 1677.07^{\circ}\text{P}^{\circ}\text{T}^{\ast}10^{-6} - 2791.5^{\circ}\text{P}^{3*}10^{-9} + \\ & 4679.44^{\circ}\text{P}^{2*}\text{T}^{\ast}10^{-9} - 1424.25^{\circ}\text{P}^{\ast}\text{T}^{2*}10^{-8} + 116.914^{\circ}\text{P}^{2*}\text{T}^{2*}10^{-9} \end{aligned}$

- $CO_2$  =  $CO_2$  equilibrium concentration in Volume
- P = equilibrium pressure (partial) in psig
- T = temperature of the liquid in  $^{\circ}$ F.

Note:

- Calculation based on beer with Specific Gravity (sp gr) of 1.01 kg/l
- Conversion from % by weight to volume:  $CO_2$  [Vol] = 5.0607 x  $CO_2$  [% by wt] x sp gr

CALCULATION OF  $CO_2$  EQUILIBRIUM CONCENTRATION according to **EBC** 

$$CO_2[\% m/m] = A \times \left(p[\text{barg}] + p_{atm}[\text{bara}]\right) \times e^{\left(C + \frac{D}{T[\circ C] + 273, 15}\right)}$$

• $CO_2$ = $CO_2$ equilibrium	concentration
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- A = conversion factor/compensation factor
- p = equilibrium pressure (partial) in barg
- p<sub>atm</sub> = atmospheric pressure in bara
- C = solubility-dependent factor (-10.74\*)
- D = product- dependent factor (2617.25\*)
- T = temperature of the liquid in  $^{\circ}$ C.

\* Calculation based on beer with an OG of 12 °Plato and apparent attenuation of 80 %

Conversion from % m/m to Volume:  $CO_2$  [Vol] = 5.061 x  $CO_2$  [% m/m] x S (specific gravity)

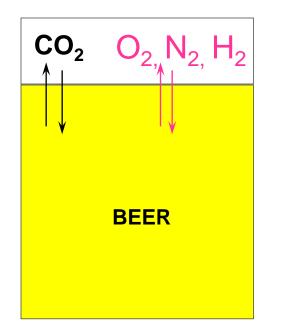
#### ERROR SOURCES MANOMETRIC CO<sub>2</sub> MEASUREMENTS

- Foreign gasses in the beverage
- Changes in the atmospheric pressure Note: Solved in most of the digital CO<sub>2</sub> meters by using a absolute pressure measurement
- Composition of the beverage

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### CO<sub>2</sub> MEASUREMENT, effect Foreign Gasses

#### CO2 MEASUREMENT based on HENRY's and DALTON's LAW



**Henry:**  $CO_2 = P * f(t)$ 

**Dalton:**  $P = [P CO_2 + P O_2 + P H_2 + P N_2]$ 

### CO<sub>2</sub> MEASUREMENT, effect Foreign Gasses

**<u>SELECTIVE CO</u><sub>2</sub> <u>MEASUREMENT OF PACKAGED BEER</u>**; calculation of CO<sub>2</sub> equilibrium concentration compensated for headspace air:

#### By ASBC CO<sub>2</sub> chart:

Calculate correction for partial pressure of air in headspace to be subtracted from the measured gauge pressure

$$P_{corrected}[psig] = \left(P[psig] + 14.7 - \frac{v_{air}}{v_{head space}} x 14.7\right)$$

By EBC:

$$CO_{2}[Vol] = A \times \left( p[barg] + p_{atm}[bara] - p_{atm} \times \frac{v_{air}}{v_{head space}} [bar] \right) \times e^{\left(C + \frac{D}{T[\circ C] + 273, 15}\right)}$$

### CO<sub>2</sub> MEASUREMENT, Failures due to Foreign Gasses

#### Supposition 1: Effect of air

O<sub>2</sub> and N<sub>2</sub> dissolved are proportional to atmospheric conditions

O <sub>2</sub> content		simulated CO <sub>2</sub> content [Vol]			
ppm w/w	0°C	5°C	10°C	15ºC	20°C
0.10	0.02	0.02	0.02	0.02	0.02
1.00	0.21	0.20	0.19	0.18	0.16

#### **Supposition 2**: Effect of Nitrogen ( $N_2$ or $CO_2/N_2$ mix-gas injection)

N <sub>2</sub> content	simulated CO <sub>2</sub> content [Vol]				
ppm w/w	0°C	5°C	10ºC	15ºC	20°C
1	0.06	0.05	0.05	0.05	0.04
10	0.55	0.53	0.49	0.45	0.42

#### CO<sub>2</sub> MEASUREMENT, Failure due to Composition of Beverage

- CO<sub>2</sub> solubility in beer 12 <sup>o</sup>Plato is approximately 0,96 x CO<sub>2</sub> solubility in water
- CO<sub>2</sub> solubility in soft drink with 10 g/l sugar (=10 <sup>o</sup>Brix) is approximately 0,90 x CO<sub>2</sub> solubility in water
- As the extract content gets smaller the CO<sub>2</sub> solubility in beer approaches the solubility in water
- Rule of thumb: approximately 1% CO<sub>2</sub> solubility per 3 <sup>o</sup>Plato or 1 <sup>o</sup>Brix

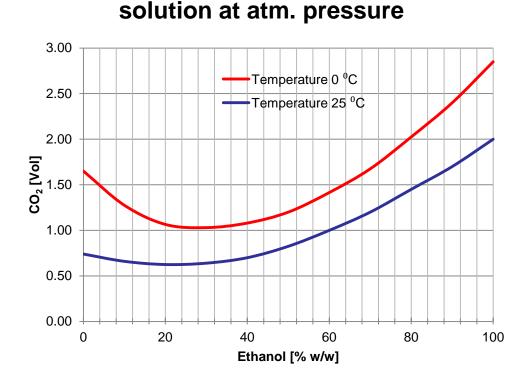
Note: In practice the difference in CO<sub>2</sub> solubility will not be taken into account

### CO2 MEASUREMENT, Influence of Ethanol Content

CO<sub>2</sub> solubility in water/ethanol

Information in literature different opinions are found :

- Paukner: in a range of 4-6 % Ethanol the CO<sub>2</sub> solubility is comparable with water
- Haffmans:



Note: In practice the difference in CO<sub>2</sub> solubility will not be taken into account

### Methods of CO<sub>2</sub> MEASUREMENT, Effects of Failures

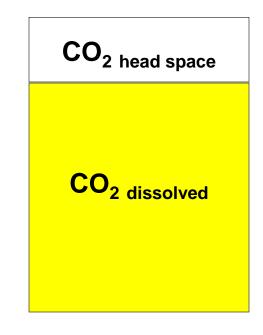
CO <sub>2</sub> measurement methods	ASBC Methods	EBC Methods	Selective	Gas/liquid	Remark
- Manometric (mechanical)					
- P&T	beer-13	9.28.3	Х	gas	
- Multi-volume expansion	Beer-13	9.28.5/6	$\checkmark$	gas	Anton Paar
- Membrane			$\checkmark$	gas	Gas selective membrane, only in-line
- Manometric (optical)					
- Adsorption			$\checkmark$	gas	only in-line
- None invasive			√ /X	gas	only bottles (glass & PET), can measure CO <sub>2</sub> pressure and total pressure
- Chemical/titrimetric					
- Blom & Lund		9.28.1	$\checkmark$	liquid	
- Titration (Corning)			$\checkmark$	liquid	
- Special CO <sub>2</sub> detectors					
- Thermal Conductivity		9.28.4	$\checkmark$	gas	Orbisphere
- Attenuated Total Reflection ATR			$\checkmark$	liquid	only in-line

#### CO<sub>2</sub> MEASUREMENT of PACKAGES



### CO2 MEASUREMENT of PACKAGES, Temp. Effects

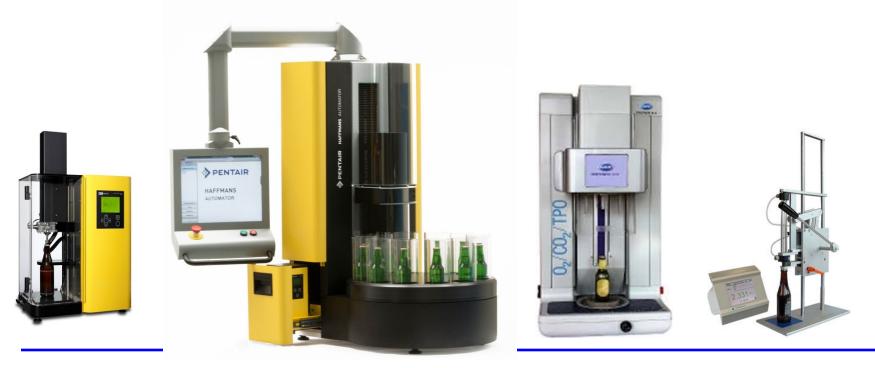
- CO<sub>2</sub> Measurement of Packaged beer (bottle, can and/or keg)
- CO<sub>2 total</sub> = CO<sub>2 head space</sub> + CO<sub>2 dissolved</sub>
- $CO_{2 \text{ total}}$  = constant and independent of temp.
- When temp. increases:
   CO<sub>2 head space</sub> (↑)+ CO<sub>2 dissolved</sub>(↓)
- Example: packaged bottle  $V_{liquid}$ = 300 ml  $V_{head space}$  = 15 ml  $CO_{2 dissolved}$  at 10 °C= 2.65 Vol  $CO_{2 dissolved}$  at 20 °C= 2.61 Vol



### CO2 MEASUREMENT of PACKAGES, Temp. Effects (cont.)

#### **Possible solutions:**

- Always measure the CO<sub>2</sub> concentration of packages on a standardized temp.
   for example 25 °C (according to ASBC method).
- Use an instrument that is able to measure the CO<sub>2</sub> concentration temperature independently: A "new" CO<sub>2</sub> quantity "CO<sub>2</sub> temp standard CO<sub>2 TS</sub>" or "normalization temp." was introduced.

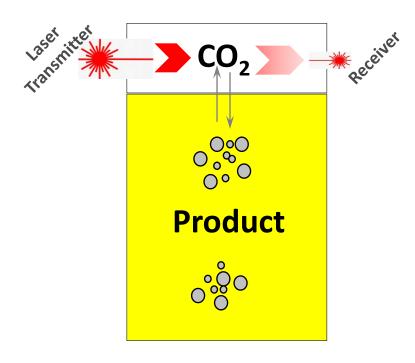


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### CO2 MEASUREMENT of PACKAGES, Non-Invasive

Next step in selective CO<sub>2</sub> MEASUREMENT of Packaged beer (bottles only)

#### **NON-INVASIVE CO<sub>2</sub> Measurement**



- Package remains unopened.
- Equilibrium pressure, Total pressure and temp. are measured without contacting the bottle.



#### Non-Invasive CO<sub>2</sub> Measurement, Video (real time)



#### 5-10 seconds for results !

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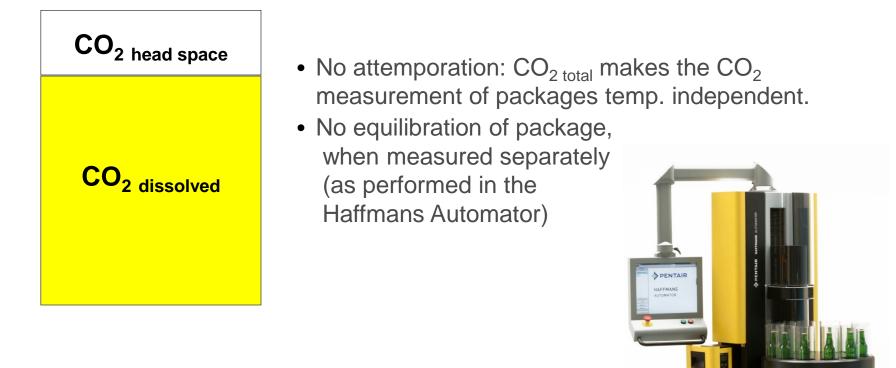
The Evolution of CO<sub>2</sub> Measurement

### CO<sub>2</sub> MEASUREMENT of PACKAGES, Next Step??

Next step in CO<sub>2</sub> Measurement of Packaged beer (bottle, can):

Analogue to TPO:

**Measurement of TPCO= CO<sub>2 total</sub> =CO<sub>2 head space</sub> + CO<sub>2 dissolved</sub>???** 



### CO<sub>2</sub> MEASUREMENT, ATR-Method (in-line)

#### New in-line CO<sub>2</sub> measurement technology is ATR.

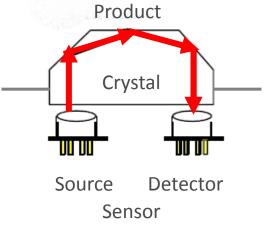
ATR Uses of infrared light (IR) and its Attenuated Total Reflectance (ATR).

Infrared beams are directed under an angle onto the optically crystal, make sapphire, that is installed at the probe's process connection and is in contact with the product.

The infrared light enters the liquid surface and through Absorption, it will attenuate and the reflected spectrum of this beam is measured.

#### ATR CO<sub>2</sub> measurement is:

- Selective
- Fast (short measurement interval) and
- Beverage composition has no influence



#### CO<sub>2</sub> MEASUREMENT CALIBRATION



### CO<sub>2</sub> MEASUREMENTS, Calibration

#### Manometric (mechanical):

- Calibration of pressure and temperature (indirect).

#### Manometric (optical):

Adsorption:

Calibration (indirect)

- CO<sub>2</sub> Differences with lab result to be corrected.

None-invasive:

Calibration (indirect) of

 total pressure and partial CO2 pressure by using a certified gas (CO2 or CO2/N2 mixture) at different pressures.

#### Special detectors:

Thermal Conductivity:

Calibration (indirect) with

Certified gas (one point or two point calibration)

#### <u>ATR</u>

- 7 fixed products stored (Beer, beverage, beverage diet, wine, mineral water, strong beer, MID) and one to create a customer specific product.
- Field calibration adjusting and aligning to trends in a repeated manner against lab result (use of offset or gain adjustment).





### CO<sub>2</sub> MEASUREMENTS, Verification

#### Trace CO<sub>2</sub>:

- An installation that produces <u>water</u> with a known CO2 quantity based on pressure and temperature conditions.

#### **High precision Calibration Kit**

An accurate determined quantity of sodium Bicarbonate is combined with a ready-made diluted Citric acid solution to provide an accurate carbon dioxide concentration only to be used to calibrate/verify the thermal conductivity sensor.



### CO<sub>2</sub> MEASUREMENT, Good Practice

- Use <u>one</u> CO<sub>2</sub> calculation, without compensating for CO<sub>2</sub> solubility, or density for all beers and beverage types at all stages of the production.
   This avoids operators involved in CO<sub>2</sub> measurement needing to adjust CO<sub>2</sub> formula's for each product which can lead to mistakes.
- At beverage development, determine preferred CO<sub>2</sub> content through taste panels. This CO<sub>2</sub> content is calculated based on the CO<sub>2</sub> calculation above.
- 3. Reproducibility of  $CO_2$  measurement is relevant for proper  $CO_2$  control, true  $CO_2$  level not relevant;
- 4. Use selective CO<sub>2</sub> measurement when foreign gas contents do not allow proper quality control.

#### What do all these CO<sub>2</sub> methods bring brewing industry

New CO<sub>2</sub> meter/measuring method have different advantages; Reduction of sample preparation/faster results Choice needs to be fine tunes with product/application Need for standardization

#### **Issues:**

Comparability of CO<sub>2</sub> measurement/methods Calibration of special detectors (ATR and Thermal Conductivity) against master



## THANK YOU FOR YOUR ATTENTION!



### Frank Verkoelen

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Pentair Haffmans