

Poster #85

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This poster describes an approach to testing the flavor quality of carbon dioxide (CO<sub>2</sub>) used in breweries. The process has been implemented in more than 100 beer production and packaging facilities, where it has contributed to a reduced risk of off-flavors, improved product quality and lower losses.

















## Introduction

The flavor quality of liquid CO<sub>2</sub> used to carbonate beer or protect it from oxygen ingress prior to packaging is of paramount importance. CO<sub>2</sub> recovered from brewery fermentation contains numerous undesirable flavors, including dimethyl sulfide, hydrogen sulfide, methanethiol and acetaldehyde. Recovery systems for this gas are designed to remove these flavors through water scrubbing, oxidation and adsorption. If such processes are not adequately monitored, they may fail. Breweries need robust sensory assessment procedures to assure CO<sub>2</sub> quality, regardless of whether they recover their own for re-use, or purchase the gas in bulk from external suppliers.

## Flavor Risks Associated with CO<sub>2</sub>

One of the biggest product recalls in the history of the global beverage industry, affecting multiple products in multiple markets, was triggered by use of contaminated CO<sub>2</sub>. Gas used to carbonate several brands of drinks was contaminated with a variety of odor-active compounds. The incident led to a dramatic drop in the company's stock price, and a loss of confidence among European consumers. This problem could, and should have been prevented.

Flavor problems associated with use of poor quality CO<sub>2</sub> are caused by a relatively small number of flavor compounds originating from a narrow range of sources as shown below.

<b>Acetaldehyde</b> <i>Acetaldehyde</i> 	<b>Earthy-green pepper</b> <i>3-Isopropyl-2-methoxy-pyrazine</i> 	<b>H<sub>2</sub>S</b> <i>Hydrogen sulfide</i> 	<b>Musty-TCA</b> <i>2,4,6-Trichloroanisole</i> 
<b>Diacetyl</b> <i>2,3-Butanedione</i> 	<b>Earthy-potato skin</b> <i>3-Isobutyl-2-methoxy-pyrazine</i> 	<b>Mercaptan</b> <i>Methanethiol</i> 	<b>Plastics</b> <i>Styrene</i> 
<b>DMS</b> <i>Dimethyl sulfide</i> 	<b>Ethyl hexanoate</b> <i>Ethyl hexanoate</i> 	<b>Mineral oil</b> <i>Various hydrocarbons</i> 	<b>Solvent</b> <i>Various compounds</i> 
<b>Earthy-compost</b> <i>2-Methylisoborneol</i> 	<b>Geosmin</b> <i>Geosmin</i> 	<b>Mushroom-moldy</b> <i>1-Octen-3-ol</i> 	<b>Sour</b> <i>Various acids</i> 

## Sampling of CO<sub>2</sub> for Sensory Analysis

The technique used to sample the CO<sub>2</sub> supply for sensory testing is of great importance. CO<sub>2</sub> gas is bubbled vigorously through odorless still water, attemperated, then served to assessors.

The following items are required: (i) Gas burner and alcohol to sanitize the sampling valve on the CO<sub>2</sub> supply; (ii) Clean, odorless plastic tubing with suitable connectors to attach to the CO<sub>2</sub> supply; (iii) A source of odorless, tasteless water, packaged in disposable PET plastic – typically a 5 liter bottle of water from which 1.5 liter of product is discarded immediately prior to use; (iv) Sample labels.

Sampling comprises the following steps:

- Fully open the valve on the CO<sub>2</sub> line for two blasts of 5 seconds each to dislodge any fluid which might be present within the gas line;
- Sanitize the connecting port on the CO<sub>2</sub> line using alcohol and flame;
- Connect the clean, odorless plastic tubing to the sanitized CO<sub>2</sub> line;
- Remove the closure from the water bottle and discard about one third of the water;
- Insert the end of the plastic tube into the remaining water contained within the bottle;
- Open the valve to allow CO<sub>2</sub> to bubble vigorously through the water. The gas flow rate should be sufficient to allow some of the water to overflow from the container during sampling (typically 2 – 2.5 liters of gas per minute);
- Continue to bubble the gas through the water for 5 minutes.
- Close the valve on the CO<sub>2</sub> main, remove the tubing from the water, and replace the closure on the water bottle;
- Take the bottle to the laboratory for sensory analysis and attemperate to 5°C.



## Sensory Evaluation of CO<sub>2</sub>

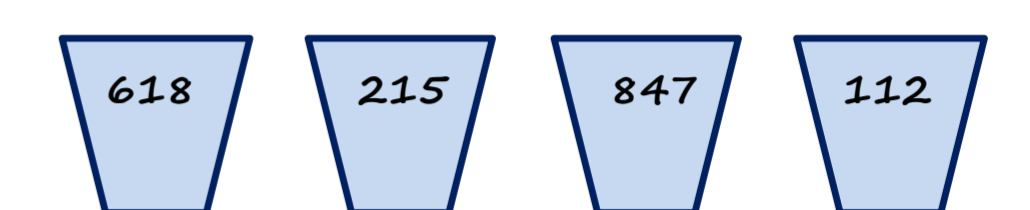
Sensory evaluation of CO<sub>2</sub> and other process gases requires the use of assessors who, at a minimum, have been trained to detect and identify the 16 off-flavors shown opposite, as well as carbonation. Training typically takes no more than a day, and comprises development of recognition skills for the 17 attributes, and practice in use of the sample assessment form.

The test procedure involves assessors rating the quality of the sample compared to their expectations and marking up to five non-conformances to justify their score. Typically waters which have been used to sample CO<sub>2</sub> are evaluated alongside those which have been used to sample air, nitrogen or oxygen. Samples (80 – 100 ml) are served in disposable plastic glasses and labelled with 3-digit random codes. To minimize the risk of presentation bias, they are served in a different sequence to each individual assessor.

Analytical Quality Control (AQC) samples are used to assure the validity of results. These comprise negative control samples (odorless water) as well as positive control samples (odorless water to which low intensity flavor defects are added in the form of food-grade flavor standards). Valid test sessions are characterized by high quality scores being generated for the odorless water samples, with lower scores for the positive controls. Replication of test and control samples provides further confidence in the performance of the test.

A useful aspect of evaluating CO<sub>2</sub> samples alongside air, oxygen and nitrogen samples is that a check can be made that samples have the expected flavor attributes. Good quality CO<sub>2</sub> should generate samples which, while free of flavor defects, are slightly sour in taste and lightly carbonated. Assessors should be trained to evaluate and recognize these features. If the sample – identified as a CO<sub>2</sub> sample – does not have them, its quality score should reflect this. This provides further confidence in the test, and a check that assessors have good capability with respect to evaluation of such samples.

### Assessment procedure



Serve samples at 5°C in random order, identified by 3-digit codes

Evaluate samples with 8 assessors

Instruct assessors to score overall quality and mark non-conformances

### Example results – sample 847

Average quality rating

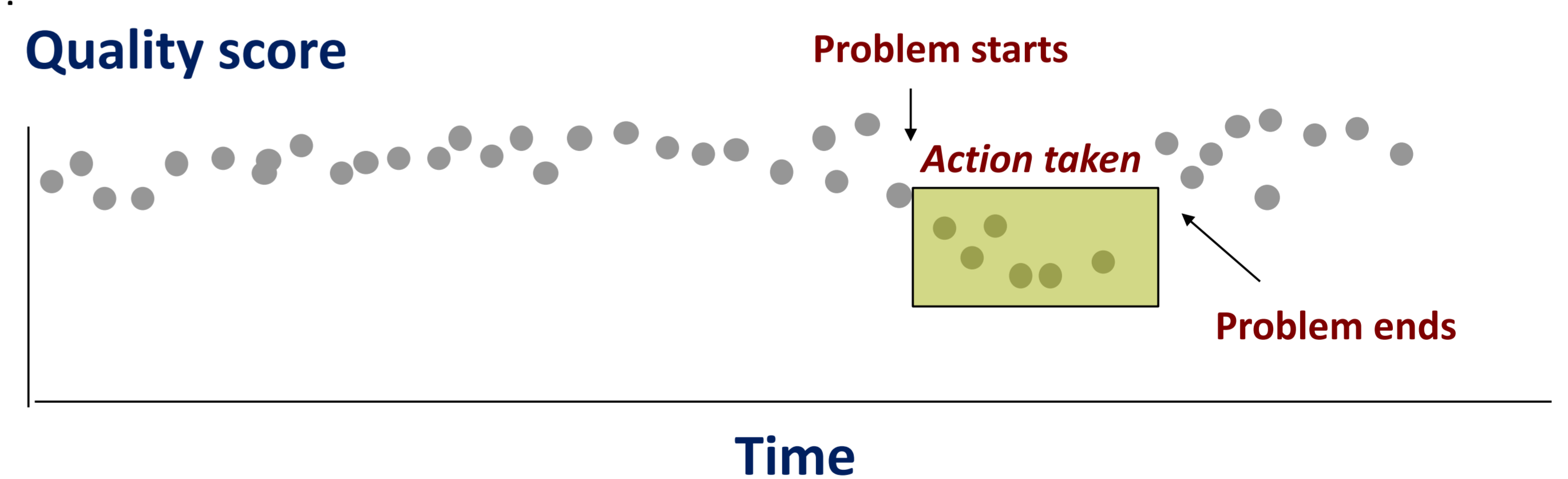
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### Non-conformances

DMS	6 assessors
Acetaldehyde	4 assessors

## Reporting, Action and Prevention of Problems

Daily quality scores generated using this sensory method can be plotted on control charts. The target is to achieve a high score, which remains stable over time. A decline in the score indicates the onset of a problem, allowing timely corrective action to be taken.



Failures in the sensory quality of CO<sub>2</sub> usually occur for the following reasons:

- Contamination of the bulk supply during transport or storage;
- Failure of the water scrubbing in the CO<sub>2</sub> recovery process, either due to overloading of the system or microbial growth in the water;
- Failure of the oxidant in the purification system, due to overloading of the system or "poisoning" of the catalyst;
- Failure to re-generate or replace the activated carbon used as adsorbent in the final stage of purification according to the schedule recommended by the supplier;
- Use of the wrong type of activated carbon in the purification system.

**Conclusion:** Flavor problems associated with use of poor quality CO<sub>2</sub> are caused by a relatively small number of odor compounds, originating from even fewer sources. By implementing the sampling and testing practices described in this poster, the chance of damaging the flavor quality of beer through use of defective CO<sub>2</sub> can be reduced.