Ministry of Environment of Denmark
Environmental Protection Agency

Climate friendly wort management in breweries MUDP-project

MUDP Report

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Miljøteknologisk Udviklings- og Demonstrationsprogram

Projektet, som er beskrevet i denne rapport, er støttet af Miljøteknologisk Udviklings- og Demonstrationsprogram (MUDP) under Miljøministeriet, der støtter udvikling, test og demonstration af miljøteknologi.

MUDP investerer i udvikling af fremtidens miljøteknologi til gavn for klima og miljø i Danmark og globalt, samtidig med at dansk vækst og beskæftigelse styrkes. Programmet understøtter dels den bredere miljødagsorden, herunder rent vand, ren luft og sikker kemi, men understøtter også regeringens målsætninger inden for klima, biodiversitet og cirkulær økonomi.

Det er MUDP's bestyrelse, som beslutter, hvilke projekter der skal modtage tilskud. Bestyrelsen betjenes af MUDP-sekretariatet i Miljøstyrelsen.

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Denne slutrapport er godkendt af MUDP, men det er alene rapportens forfatter/projektlederen, som er ansvarlige for indholdet. Rapporten må citeres med kildeangivelse.

Contents

1. Preface and background

1.1 Foreword

The present project concerns the investigation of the possibility of shortening the boiling time in beer brewing without adversely affecting the quality of the beer. This is achieved by stripping the hot wort with carbon dioxide.
The partners in the project are Randers Bryghus, Humleland, The Scandinavian School of Brewing, Maskinfabrikken Hillerslev and Frank Innovation. Frank Innovation has been project holder and project manager. In addition, IN-Water and SDU have participated as sub-consultants, and Thisted Bryghus has made experimental facilities available.

The project has been completed in the period 2020-2024.

For the sake of readers without a professional background in the brewing industry, a brief explanation of the structure and function of breweries is given.

1.2 Structure and function of breweries

Beer is brewed in basically the same way in all breweries, and barley malt is the primary raw material and extract agent.

The malt is produced in malthouses where barley is germinated. Enzymes are thereby formed, and the barley's starch grains are made available. After germination, the malt is dried in a kiln. For most types of malt, the drying takes place at a high temperature, but pilsner malt and other light malt types are dried gently keeping enzymes preserved.

The brewing process begins with malt being crushed in a mill. The crushed malt is taken to a mash tun, where there is hot water, here the mash goes through a time/temperature program, where the malt's enzymes convert its starch into sugars, primarily maltose. After removing shell parts etc. in a lauter tun (a mash separator), the maltose-containing liquid mixture – the wort – is transferred to a wort kettle, after which the wort is boiled for a good hour. Hops are added during boiling, and bitter-tasting alpha and beta acids are formed, which are slowly released into the wort. During boiling, the off-flavor substance dimethyl sulphide (DMS), which is volatile, is removed. The wort contains small amounts of other foul-smelling substances, which are also removed to varying extents by boiling.

DMS originates from the malt's amino acids, which also contain readily soluble precursors to DMS that are not volatile. These precursors are slowly converted to DMS at high temperature, which is why the brewing time of the wort is conditioned by the fact that both DMS and precursors to DMS must be removed to a level so that DMS cannot be tasted in the finished beer. This applies particularly to brews with a lot of pilsner malt and other light malt types. After boiling, the wort is fed tangentially to a whirlpool, where precipitation - trub - collects on the bottom, while the clarified wort is led to cool down and then to a pressure tank, where it ferments and is matured. The fully matured beer is then taken to filtering or centrifugation before bottling.

The processes in the breweries' brewhouses are energy-intensive, and it is especially the wort boiling that consumes a lot of energy. An estimated 20-30% of the breweries' thermal energy is included in this.

1.3 Brewing using BAT technologies

In order to save energy and the emission of CO_2, different technologies can be used in connection with the wort boiling. In the EU, BAT technologies are defined, which are the best available technologies that the large European breweries must implement.

It is BAT technology (BAT 18) to increase energy efficiency by:
1) mashing in at approx. 60°C, which reduces the use of cold water
2) decrease in the evaporation rate to approx. 4%/h during the wort boiling, e.g. by two-phase boiling and dynamic low-pressure boiling
3) to increase the share of HGB/degradation, i.e. production of concentrated wort, which reduces the amount to be boiled, so that energy is saved.

The methods are used in virtually all large breweries, also outside the EU.

1.4 Development of technology for climate-friendly brewing

In 2020, under the MUDP program, the present project - Climate-friendly Wort Handling in Breweries - was initiated to investigate the possibilities of limiting the breweries' usual boiling time for the wort boiling of 1 - 1½ hours.

The basis for the project was knowledge of the existence of equipment (Krones/Steinecker) for removing DMS from hot wort just before cooling. That is when the wort has been passed through the whirlpool and possibly further cleaned in cartridge filter or similar. The process consists of carbon dioxide mixed with nitrogen and air being passed over a falling film of wort at boiling temperature, whereby DMS evaporates into the gas mixture. The project's working hypothesis was that pure carbon dioxide can be fed directly into the whirlpool, whereby extra equipment to remove DMS can be saved.

The project has been affected by the corona shutdown, sickness and project changes.

This report gives an account of the work carried out. The report is divided into sections that broadly follow the project description with several changes agreed with the Danish Environmental Protection Agency.

Thanks to
• Thisted Brewery for shelter and assistance when testing new technology
• Ultraaqua for help in developing a method for the analysis work
• Carlsberg's Research Laboratory for analytical work
• Harboe Bryggeri for the supply of wort concentrates
• MS Slagballe Brewery for delivery of fresh wort
• Gert Holm Kristensen from IN-Water and Professor Frants Lauritsen from SDU. They have been subcontractors, but their help has gone far beyond that.

2. Summary and Conclusions

Literature studies and test work
A literature survey provided a thorough assessment of the conditions concerning DMS in wort and beer. In addition, necessary design data was provided for the construction of a test whirlpool. It was surprising that there is quite a large variation in the determinations found in the literature for the rate constant when converting DMS precursors to DMS.

A further literature survey aimed at stripping processes provided a further understanding of the problems surrounding aroma substances in wort and beer.

Sensory and chemical analyses
In Denmark it is not custom to analyze for DMS, which is why it was intended to analyze/assess DMS using sensory analysis. This proved to be difficult.

With knowledge of measurements of volatile substances in swimming pools (Ultraaqua) with a MIMS method developed by SDU, we started a collaboration with SDU on DMS measurements. When measuring with MIMS - Membrane Inlet Mass Spectrometry - the sample diffuses through a membrane, and components that diffuse through the membrane are analyzed via mass spectrometer - sorted by mass.

In connection with the project, SDU has documented that the method can be used and that DMS can be measured at-line with a fifteen-minute delay. However, the MIMS method does not determine precursors, for which reason Carlsberg's Research Laboratory was contacted. The laboratory has performed analysis of DMS and Total DMS, where Total DMS includes precursors.

Virtual test whirlpool
The intention was to develop a virtual test whirlpool, because at the start of the project there was no rapid method for measuring DMS, but since SDU could measure DMS at-line during the experiments with a test whirlpool, we chose to focus on increasing the number of analyses instead. This decision was also justified by the fact that developing a virtual test whirlpool turned out to be more demanding and costly than expected.

Test whirlpool
Maskinfabrikken Hillerslev constructed a test whirlpool of 400 liter. The design was according to the instructions in the literature survey, and it has worked perfectly. The whirl pool was placed at Thisted Bryghus.
Thisted Bryghus is not a partner in the project, but it turned out that the smaller breweries generally do not have problems with DMS. The reason is the physical conditions. DMS is volatile and mostly disappears by direct evaporation at high temperature. All wort kettles have a surface and a volume primarily given by the volume they contain, but the surface is determined by the radius in the 2nd power and the volume is determined by the radius in the 3rd power. That is, in smaller breweries there is a relatively large surface and thus a correspondingly large evaporation of DMS. In larger breweries, there is a relatively small surface and a correspondingly small evaporation of DMS. - DMS is primarily the big breweries' problem. Based on the above, we received the grantor's permission to set up the test whirlpool at Thisted Bryghus.

The project's experimental work
A large number of tests have been carried out at Thisted Bryghus, partly in a test cylinder and partly in the test whirlpool. The production conditions at Thisted Bryghus are such that, in connection with the wort boiling, wort can be led directly into the test whirlpool. Thisted Bryghus brews several pilsner brews in series, which has made it possible to carry out experiments with very similar brews.

The tests carried out provided a good basis for full-scale trials at Randers Bryghus and the final documentation of the experimental plant at Randers Bryghus and Humleland in Aarhus.

While all the experimental work in the test column and test whirlpool during the experimental work has been carried out with pilsner beer, the final documentation has included work with different types of beer to get a broad overview of the application possibilities of the developed technology. All experiments with the test whirlpool and the initial full-scale experiments at Randers Bryghus were carried out with ½ hour's boiling.

Screening experiments with different types of beer have been carried out with 40 minutes of boiling.
Planning for the final full-scale trials at Randers Bryghus and at Humleland has taken place, so there has been great certainty that the finished beer has been fully satisfactory in terms of quality.

Conclusions
• The project has resulted in the development of a rapid method for analyzing DMS.
• It has been documented that the boiling time by wort boiling very likely can be reduced to ½ hour for lager-type beers and to 40 minutes for most other types of beer.
• It is probable that the method used for stripping the hot wort gives the produced pilsner-type beer a fresher aroma and taste. This without the hop aroma and other taste parameters being affected.
• The project's results have formed the basis for Frank Innovation, together with Thisted Bryghus and Hillerslev Maskinfabrik, to have applied for and been granted MUDP funds for an ongoing project “Wort Boiling without Boiling”, where the limits of a stripping technology are being investigated. Partly with the wort line as a reactor, where the carbon dioxide is blown into the wort line and partly by blowing carbon dioxide directly into the wort boiler.

3. Literature study and analytical methods

The chapter deals with knowledge gathered in key areas of particular importance for the effective implementation of the project. Partly the formation and removal of the unwanted volatile substance DMS in the brewing process is explained, and partly a new method for at-line measurement of DMS that has been identified is described. In addition, the focus is on the function of the central process - the brewery's whirlpool - where suspended matter - hot trub - is separated from the wort before the fermentation process. The process is central to the current project, because the stripping of DMS must be done without disturbing the sedimentation of hot trub.

3.1 Dimethyl sulphide DMS

Dimethyl sulphide is a colorless liquid that catches fire in open air. It is soluble in water and in wort. The primary use of DMS is in the perfume industry, for which a 0.1% solution of DMS is sold. The structure is shown in FIGURE 3.1.

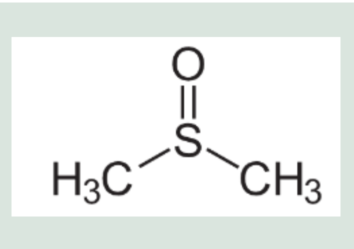

FIGURE 3.1. Structural formula for DMS.

As with many other sulfur compounds, DMS has an unpleasant odor and taste.
In general, the smell and taste of DMS in beer is considered a fault, but there are light beer types where a weak DMS smell and taste is included in the description. Perhaps because it historically has been difficult to avoid.

The recognition of DMS in wort and beer is difficult. Smell and taste are described as "cooked vegetables", "canned corn" and "cabbage". Smells and tastes that can also be described as "un fresh" in low concentrations. Imagine you sit with your favorite lager and think "well", maybe I try a different brand next time. The reason for the difficulties is that many substances naturally found in beer can mask the recognition of DMS. Substances such as phenyl ethanol and phenyl acetate and phenylethyl acetate can mask the recognition of DMS. Linalool (hop aroma) as well as ethyl acetate and isoamyl acetate also have an influence.

The threshold value for recognition of DMS is stated in many sources to be in the range of 25-150 µg/l. This is very broadly consistent with the recognition of DMS being difficult.

3.1.1 Occurrence of DMS in the brewing industry's processes

During malting of barley, DMS is formed from the precursor S-methyl methionine (DMS-P), which is named SMM. SMM is not volatile and it is easily soluble in water and wort. Barley's SMM content depends on the barley variety and the barley's nitrogen content, etc.

During the drying of malt, the oxidation product dimethyl sulfoxide (DMSO) is formed from DMS. DMSO is easily soluble in water and it is not volatile in connection with the wort boiling.

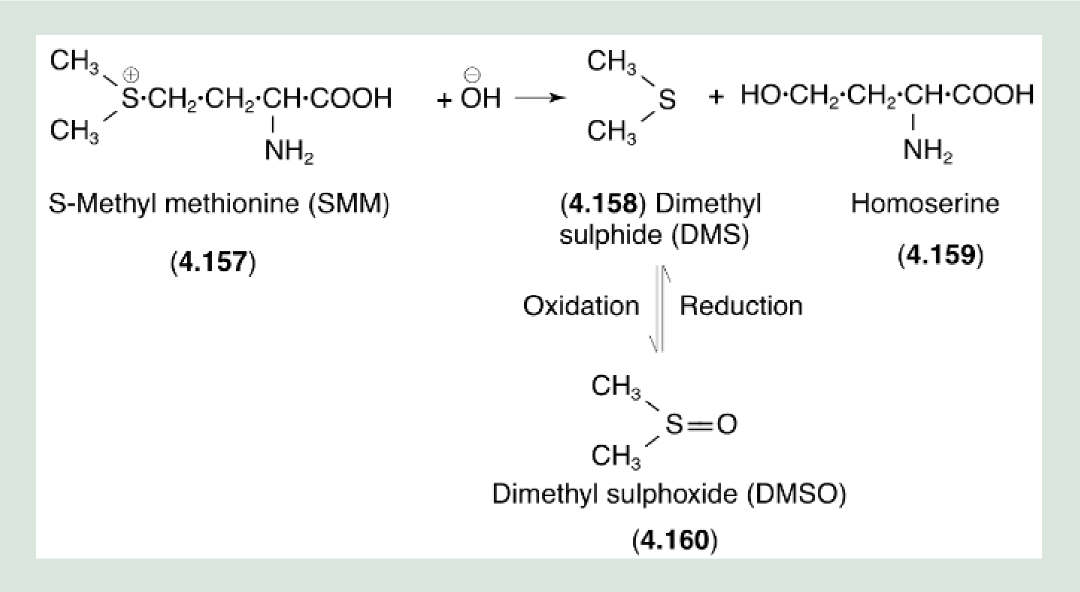

FIGURE 3.2. Formation of DMS and equilibrium with DMSO, (The Scandinavian School of Brewing).

At temperatures above 70-75°C, SMM breaks down, as indicated in FIGURE 3.2, to DMS by a 1st order reaction. The half-life at 90°C is 236 minutes, at 100°C it is 80 minutes and at 110°C it is 22 minutes, as can be seen from FIGURE 3.3.

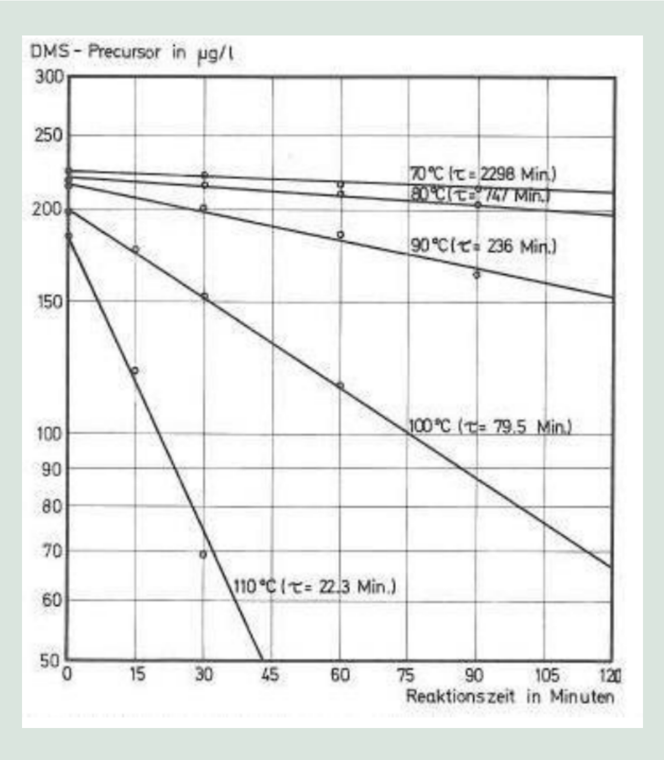

FIGURE 3.3. Degradation of DMS-P to DMS, (Krottenthaler et al. 2010).

Drying of dark malts takes place at such a high temperature that a significant part of SMM is converted and DMS subsequently evaporates. When drying light malts such as pilsner malt, the temperature is so low that a lot of SMM remains in the malt, and there is a significant amount of free DMS in the malt.

There will usually be many times more SMM than DMS in light malt. For a brewer, it is therefore most important to know total DMS (MMS plus DMS) in both malt and wort kettle.

During drying, DMS also forms an oxidation product dimethyl sulfoxide (DMSO) - see FIGURE 3.2. It can be reduced to DMS by wort bacteria. However, DMSO is not a problem under normal production conditions with effective cleaning.

During mashing, DMS and SMM dissolve in the wort. Some DMS evaporates, but usually no conversion to DMSO occurs.

During the wort boiling, SMM is converted to DMS, which evaporates or is stripped out with the steam bubbles. Depending on the intensity of the boiling, there is very little DMS left at the end of the boiling. The amount of SMM that remains depends solely on the length of the boiling time and the temperature at the time of boiling. Thus, the amount will be halved by boiling for 80 minutes at 100°C.

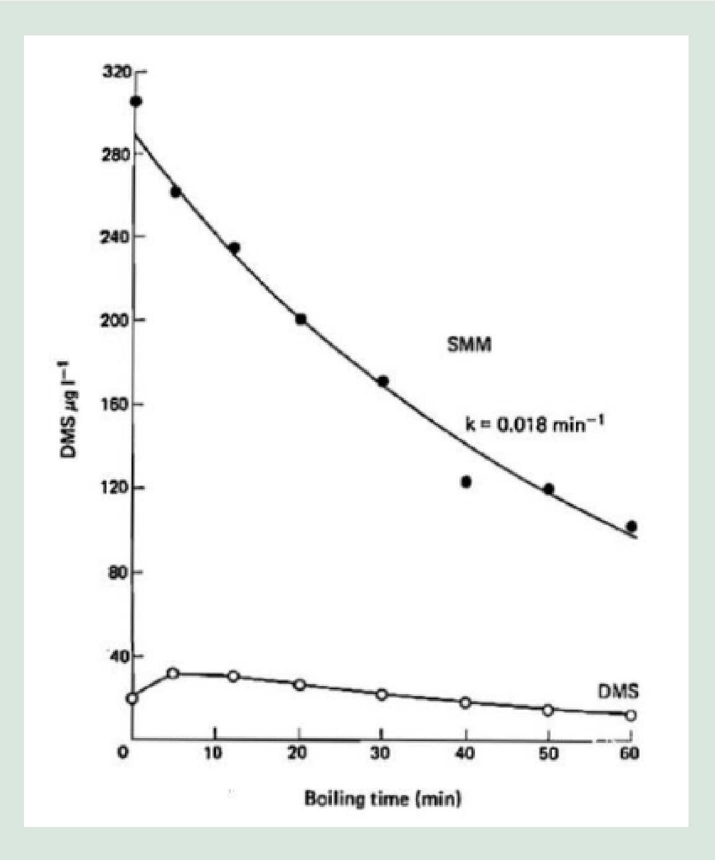

FIGURE 3.4. Concentrations of SMM and DMS during wort boiling (Wilson & Booer, 1979).

After pumping into the whirlpool, there is no longer any driving force to remove the DMS that is formed, which is why, broadly speaking, a choice must be made between a long boiling time, which gives low DMS in wort and beer, and the uncertainty associated with a rapid completion of the process in the whirlpool.

There are a large number of technical solutions that can deal with the problem, partly by de facto raising the boiling temperature using special boiling techniques or by handling DMS in hot wort with stripping to carbon dioxide after the stay in the whirlpool. They are all costly.

FIGURE 3.5 below shows an example of a 10-minute difference in boiling time.

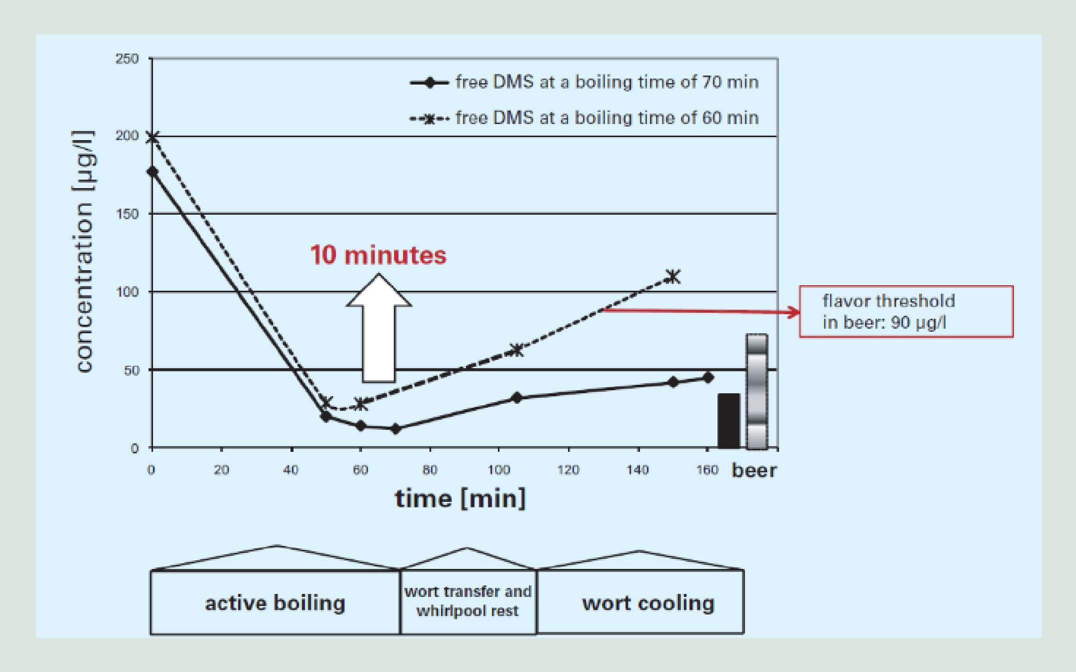

FIGURE 3.5. Free DMS at wort boiling, whirlpool and cooling (Rübsam et al., 2010).

In connection with the fermentation, the amount of DMS will decrease, as a proportion will be stripped out with the carbon dioxide formed during the fermentation. This stripping can be enhanced by fermentation at high temperature, which, however, can obviously have other disadvantages.

For a more in-depth review of the conditions surrounding DMS, please refer to Appendix 1, where the Scandinavian school of Brewing supplemented by Virtual Water Technology provides an in-depth description supplemented by a long bibliography.

3.2 Literature on Whirlpool

As described in Chapter 1.2 Structure and function of breweries, removal of hot trub takes place in a Whirlpool, where the wort is introduced tangentially and where the wort settles in a cake in the middle of the flat bottom. This technology was introduced around 1960 to replace the cools-hip or hop filter of earlier times.

Coolship, which is a flat-bottomed vessel, serves both to deposit the trub and to cool the wort. The disadvantages of coolship are that there is no heat recovery and that the wort is easily infected. The disadvantage of hop filters is primarily that they are sensitive to clogging, and that therefore a large proportion of hop leaves must be included when hops are added, and functioning as a filter aid.
The author of this paper has constructed a hop filter for a 10 hl brewhouse, it worked fine, and he has seen a coolship in operation at the Drie Vontainen (spontaneously fermented beer) brewery in Brussels. It was very strange to see.

Around 1960, the whirlpool technology was introduced. Due to the efficiency in removing hot trub, and due to savings in labor and due to improved possibilities to recycle the heat, it quickly became the dominant technology.
As described in Chapter 3.1 Literature on Dimethyl sulphide – DMS, the consequence was that, to some extent, beer with a taste of DMS/boiled vegetables/canned corn was sold.

Perhaps the whirlpool technology has both contributed to the growth in the size of the breweries and bad taste in a proportion of the beer and thus also to the "Beer Revolution" around 1990/2000?

3.2.1 Hot trub

The precipitations that occur during the wort boiling can be fairly effectively removed in a whirlpool, if simple design rules are followed. In contrast to cold trub, which is precipitated during fermentation, hot trub has large particle sizes (20-80 µm) and they have twice the density of cold trub. The hot trub consists of proteins, polyphenols and other organic substances, bitter substances and minerals.

Precipitation of hot trub is most effective if a portion of the mash is boiled (decoction). Low pH and long boiling time are also advantageous. The purpose of this project is to save energy, which is why, in connection with the experiments, attention has been paid to the deposition of hot trub in the whirlpool.

3.2.2 Whirlpool design and operation

Whirlpools are often constructed as shown schematically in FIGURE 3.6. The cooked wort is pumped in tangentially at a speed that initiates a rotation in the wort. After a pause of 20-30 minutes, the hot trub is collected in the middle of the flat bottom - in the same way as seen in a teacup with tea leaves.

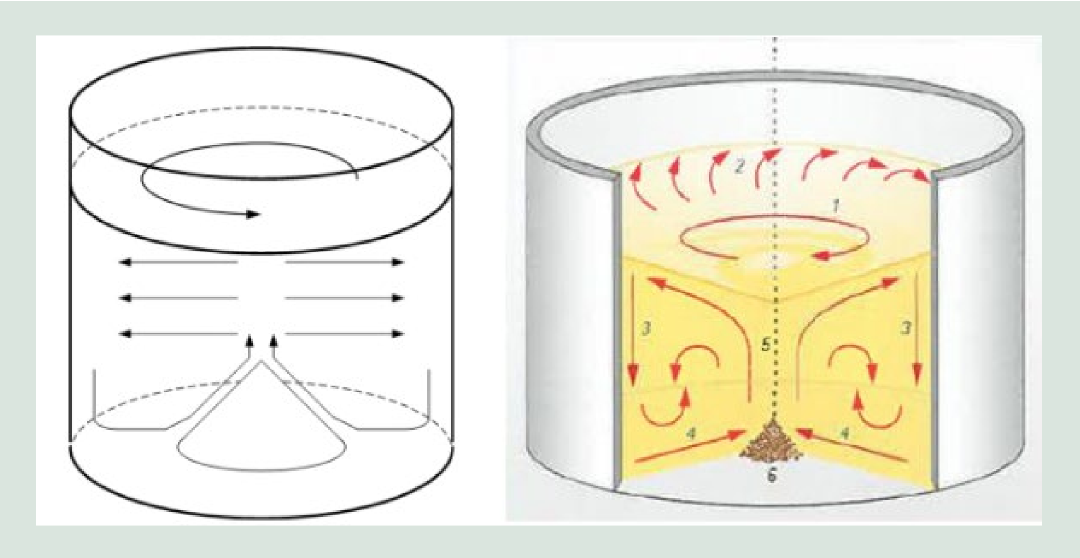

FIGURE 3.6. Circulation flow pattern (Kunze, 2019).

A number of narrow guidelines apply to the construction, and if these are followed, the whirlpool will also function as intended:

• Optimal height/diameter ratio is 0.7:1 or 0.8:1. It can be up to 1/1 if the volume is small (the trub has a short path to the bottom)
• A flat bottom with a 1% slope towards a bottom outlet.
• The tangential incoming flow must have a speed of 3-3.5 m/sec, and the incoming piping must have an angle of approx. 17° pointing into the whirlpool.
• The inlet must be approx. 1/3 of the way up the vessel, typically around 1 meter in a large whirlpool.

A number of other designs exist, the above being the most common.

As stated in Chapter 3.1 on DMS, the residence time of the wort in the whirlpool should be limited as much as possible to avoid inappropriate formation of DMS. This means that filling and precipitation of the trub should take about half an hour, and that the cooling should take place within an hour. In practice, however, it often lasts longer.

In Appendix 1, prepared by the Scandinavian School of Brewing, a thorough description of how a whirlpool should be constructed, what the flow pattern is like and how it is operated is given. In addition, a long literature list on the subject is provided.

3.3 Analysis for DMS

DMS and DMS Total (DMS plus precursors) are not analyzed at laboratories in Denmark, and the analyzes at the German laboratories are relatively expensive. In addition, a time factor is included when using external laboratories. The project was therefore planned with sensory evaluation as a guiding tool, so that several tests could be carried out in quick succession.

More precise experiments also had to be carried out with analyzes of DMS and precursors done at an authorized laboratory. There was also the planned development of a virtual whirlpool, where the formation and removal of DMS could be described and virtual experiments carried out, see 3,4 Virtual Whirlpool.

3.3.1 Sensory evaluation

After a number of sessions of tasting different concentrations of DMS in wort, we came to the conclusion, that the approach was not realistic as a general tool in this project. Even for trained tasters it was exceedingly difficult. We tried different approaches, and found that the taste and aroma of DMS shifted depending on the matrix chosen (wort, brewed beer, NA beer etc). This is in accordance with the information in Chapter 3.1 Dimethyl sulphide - DMS.
The material on the evaluations is attached in Appendix 2.
Hence it was decided, that sensory evaluation should be used as a tool for describing the finished beer flavor only.

3.3.2 Rapid method for determining DMS

At the beginning of the project, we knew that the company Ultraaqua uses a rapid method for determining volatile components in swimming pool environments. The rapid method is Membrane Inlet Mass Spectrometry - MIMS - for the analysis of gases and volatile organic substances in liquids and air. It is based on a polymer membrane that acts as the only separation between the sample and a small portable mass spectrometer. The sample's volatile substances dissolve in the membrane, diffuse through it and are ionized in the mass spectrometer, where they are analyzed by mass. In this way, it is possible to continuously follow variations in the chemical composition of the sample.

At Ultraaqua, we have tested the method on wort and added different amounts of extra DMS to wort, which gave a satisfactory addition curve with no signs of interference from other components in the wort. Further investigations confirmed our view, but during the final tests, Ultraaqua experienced noise, which made us contact Mikrolab, who supplied the equipment, and then Professor Frants Lauritsen at SDU, who originally designed the equipment.

At SDU, the method and equipment were adjusted so that it better suited the purpose, and an agreement was reached on the delivery of analyzes for the project. We also made an agreement with SDU about at-line analyzes at Thisted Bryghus, while we carried out experiments with the test whirlpool. It was super effective.

FIGURE 3.7. Analysis of DMS at Thisted Bryghus. Frants Lauritsen (SDU) and Else Birk (Frank Innovation).

Appendix 3 contains an explanation by Professor Frants Lauritsen of how the analyzes were carried out.
A detailed description of the MIMS methodology can be found in the article “Membrane inlet Mass Spectrometry; Kotiaho T. & Lauritsen F. (2002).

Recognizing that there is also a need to know the amount of precursors to DMS, we contacted head brewer Erik Lund at Carlsberg's Research Laboratory. During the course of the project, they measured DMS and total DMS (DMS plus precursors) on selected samples.

Both the SDU analyzes and the Carlsberg analyzes show quite a large spread in double determinations, and the analyzes from SDU are systematically at a slightly higher level than the analyzes from Carlsberg. Since we are examining the effects of given measures, this difference is considered irrelevant in the present context.

3.4 Virtual Whirlpool

In collaboration with Virtual Water Technology ApS, preliminary work has been carried out with a view to obtaining the necessary knowledge to be able to set up a CFD model - Computational Fluid Dynamics - for a virtual whirlpool. The advantage of establishing a mathematical model – a virtual whirlpool – will be that it will be possible to carry out an optimization of the whirlpool's design when it is to be operated with the blowing in of CO_2 bubbles with the resulting disturbances of the usual flow pattern.

Formation and removal of DMS in the wort in the whirlpool is complex and affected by sub-processes.
Formation of DMS is determined by the temperature of the wort and the concentration of precursors to DMS, while removal is determined by the mass transfer from liquid phase to gas phase – either through the liquid surface in the whirlpool or through the surface between the wort and carbon dioxide bubbles.
The temperature and concentration distribution in the whirlpool will also be determined by the flow pattern of the wort in the whirlpool, and this pattern will also affect the transport of carbon dioxide bubbles. Finally, the flow pattern, and thus the transport and sedimentation of hot trub, will also be affected by the movement of the bubbles. All in all, a complex problem, where a virtual model will be able to facilitate the experimental work considerably.

In order to gain insight into the above-mentioned multiple and inter-linked effects, it was planned from the start that the project should establish a CFD modeling of the whirlpool, where the transport of heat, suspended matter and bubbles are taken into account.

In the model, the whirlpool is divided into small geometric units, in which the systems of equations are solved numerically. This provides a description of the distribution of liquid speed, substance concentration, bubble concentration and temperature in the whirlpool. The model is thought to be primarily used to describe the formation and removal of DMS as well as the transport/settling of trub particles.

The idea was that the project's experimental work could determine a number of process parameters that could be used as input to the model (e.g. DMS formation rate and liquid to gas mass transfer rates, etc.). After such a calibration of the CFD model, the model had to be used to carry out virtual tests of design and operating strategies in order to gain insight into the meaning of the process' operating variables.

A literature review was made, and a list of necessary/desired parameters for a model was prepared. In addition, presentations were prepared for 4 experiments, which aimed to:
- Establish concentrations of SMM (S-methyl Methionine) in wort and to establish the rate constant for the formation of DMS at constant temperature.
- Establish an understanding of the dynamics during bubbling of wort with carbon dioxide.
- Establish rate constants for degassing DMS from wort as a function of the bubbling speed.
- Estimate the bubble size at 3 selected bubbling-through speeds.

The literature review is attached as Appendix 4. It should be noted that significant elements of the literature review are included in Literature Survey Appendix 1 from Scandinavian School of Brewing.

We subsequently had to recognize the complexity of the task in relation to the funding that was available, and therefore opted out of further work with a virtual whirlpool in the project. This is also because - based on positive initial experiences - we had started a collaboration with Professor Frants Lauritsen, SDU on the development of the MIMS method for rapid analysis of DMS, which would make it possible in the project to shed light on the very central kinetics of DMS formation in the wort.

4. Testwhirlpool – construction and testing

The project's test whirlpool was designed based on the extensive knowledge collected and with particular consideration of the interaction between stripping and settling of hot trub. The function of the test whirlpool was clarified through a series of experiments, where different operating conditions for dosing carbon dioxide – quantity and time – were tested for a number of brews. The tests were primarily based on Thy pilsner, where the duration of the wort boiling was varied. The nature of the hot trub cone was assessed and the stripping of DMS monitored to clarify the interaction between the processes. The method used for supplying carbon dioxide could halve the DMS content of the finished wort with a satisfactory hot trub cone, while the wort's boiling time was reduced to 30 minutes.

4.1 Basis for the construction of the test whirl pool

It appears from literature review from the Scandinavian School of Brewing (Appendix 1) that whirlpools are constructed with a relatively low height due to precipitation of hot trub. Often with a height/diameter ratio of 0.7. The ratio can be changed up to 1 if the whirlpool is small. We chose to construct a test whirl pool of 400 liters with the same height and diameter. In addition, we followed the guidelines in the literature review.

Placement of the nozzle for injecting carbon dioxide was the subject of some consideration and discussion. In light of this discussion, we chose to place the nozzle immediately before the inlet to the whirlpool.

Based on the total volume of 400 liters, we decided on an effective filling of 300 liters. The vessel was insulated and provided with a loosely insulated lid. An electric heating line was inserted to be able to maintain the temperature throughout the process. The test whirlpool is illustrated by photos in FIGURE 4.1-4.3.

At the picture FIGURE 4.2, two outlets are seen on the side as well as a bottom outlet, which is placed at a gently sloping bottom. Above the green hose is a tangential inlet with a 17-degree angle to the tangent.
As can be seen in picture FIGURE 4.3, there is an effective disposal of hot trub. The brew tested here was heavily hopped – hence the significant hot trub cone.

The initial testing of the test whirlpool did not offer any major surprises. Mechanically, it worked optimally. Under normal operating conditions, the deposition of hot trub worked fine. We chose a set-up just below the wort kettle with a direct inlet to the whirlpool, which meant that we could fill it during the 15-20 minutes it normally takes to pump from the wort kettle to the whirlpool at Thisted Bryghus. With the chosen location, we could work on the experiments without disturbing production, and samples for analysis could be taken without difficulty.

FIGURE 4.8. Test whirlpool at Thisted Bryghus.

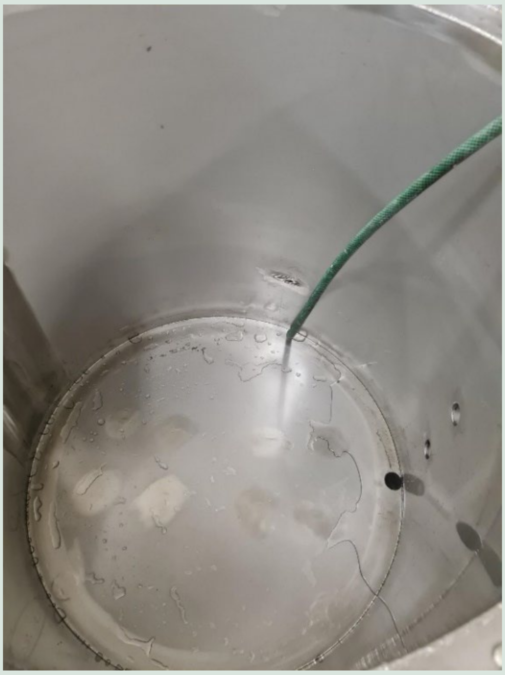

FIGURE 4.2. Test whirlpool, inlet and outlet.

FIGURE 4.3. Test whirlpool with trub deposit.

4.2 Initial tests with test cylinder

On the basis of the collected literature and professional discussions, we reached the conclusion, as stated in Chapter 4.1, that the carbon dioxide should be injected directly into the hot wort as close as possible to the inlet to the test whirlpool. We agreed to start with simple experiments in a test cylinder holding 25 liters, which was available from a previous MUDP project on de-oxygenation water in connection with high-gravity brewing. Photo of the cylinder is shown in FIGURE 4.4.

FIGURE 4.4. Test cylinder for initial trials.

A number of experiments were carried out with the aim of shedding light on the significance of variations in carbon dioxide flow - the way of thinking was that we had to keep DMS constant or slightly decreasing in the whirlpool. The thinking was also that the amount of carbon dioxide used should be so small that it would not interfere with the deposition of trub as the carbon dioxide bubbled up the wall of the whirlpool. The theory/thought was further that transverse turbulence should help to carry DMS out to the layer of carbon dioxide bubbles.

The experiments showed that flotation does not occur when bubbling through with carbon dioxide, and that a significant carbon dioxide flow must be used to control the DMS concentration.

4.3 Trials with Testwhirlpool

The experiments in the test whirlpool were carried out in direct continuation of the experiments in the test cylinder. Therefore, consecutive numbering is used for the experiments in the test cylinder and in the test whirlpool. In the last experiments, Professor Frants Lauritsen made at-line measurements. In the other experiments, samples were taken for subsequent analysis at SDU.

Operationally, we chose a run-in time of 15 minutes for the experimental work. - 15 -20 minutes is normal. At Randers Brewery, it is 10 minutes. Due to friction and a small mass, it was necessary to recirculate the wort with a pump to mimic the conditions of a large whirlpool. We chose to recirculate for 30 minutes. Drainage from the whirlpool was one hour, which is also equivalent to usual operation.

4.3.1 Experiment with Brown Ale

The purpose of the experiment was:
- to clarify the difference between normal boiling for 60 minutes (Brew 6) and boiling for 30 minutes – (Brew 7),
- to demonstrate the efficiency of the whirlpool for beers with relatively high hops,
- to shed light on the DMS conditions in a beer with a relatively very dark malt.

As can be seen from FIGURE 4.5 and FIGURE 4.6, the trub cones are high for both Brew 6 and Brew 7 - and relatively firm. Boiling time of 30 minutes results in a slightly looser trub cone. The height of the trub cones is 8 cm.

The DMS values found are seen in TABLE 4.1. As expected, low DMS values and an increase in the DMS values are seen due to the conversion of precursors to DMS.

FIGURE 4.5. Brew 6 – 60 minutes of boiling.

FIGURE 4.6. Brew 7 – 30 minutes of boiling.

TABLE 4.1. Data for measured DMS concentrations from experiments with Brew 6 and Brew 7.

Sample	**DMS Brew 6 60 min boiling**	**DMS Brew 7 30 min boiling**
Trial time	**ppb**	**ppb**
Start recirkulation	9	11
End recirkulation	27	28

4.3.2 Experiment with Thy pilsner to clarify the importance of the various parameters

As stated during the experiments with the test cylinder, we expected to shed light on conditions with low carbon dioxide flows. Based on the same way of thinking, in the first experiments carbon dioxide only was added during the recirculation. In experiments 8 -11, it was investigated whether carbon dioxide can be added at the same time that trub must be deposited.

Brew 8 and Brew 9

Brew 8 and Brew 9 show the difference in the trub formation with a moderate (2½ l/min) supply of carbon dioxide during the recirculation compared to a brew where no carbon dioxide was added. As can be seen on Brew 8, the supply of carbon dioxide gives a loose trub cone. DMS was not measured in these experiments.

FIGURE 4.7. Trub cone during experiments with Brew 8.

FIGURE 4.8. Trub cone during experiments with Brew 9.

Brew 10 and Brew 11

Experiments with Brew 10 and Brew 11 highlight the difference by limiting carbon dioxide dosage to the first 15 minutes of the recirculation. Brew 10 is carried out with a carbon dioxide dosage of 7.5 l/min during 30 minutes of recirculation and Brew 11 is carried out with a carbon dioxide dosage of 7.5 l/min during the injection and during the first 15 minutes of the recirculation. DMS was only measured in the experiment with Brew 10.

The trub deposition in these experiments is illustrated in FIGURE 4.9 and FIGURE 4.10. The cone at Brew 10 was slightly flowing with a height of around 2½ cm, while at Brew 11 it was firm with a height of 3.5 cm. The experiment shows that the remaining 15 minutes of recirculation without carbon dioxide dosing is sufficient to lead to deposition of a satisfactory tube cone.

FIGURE 4.9. Trub deposition in trials with Brew 10.

FIGURE 4.10. Trub deposition in trials with Brew 11.

TABLE 4.2. Data for measured DMS concentrations from experiments with Brew 10.

Sample	DMS Brew 10
Trial time	**ppb**
Start inlet	15
End inlet	20
Recirkulation	15
Start discharge	17
Mid discharge	23

The relatively low DMS values in the experiment with Brew 10 are shown in TABLE 4.2. As expected, they show a slight increase during the discharge.

Brew 12

Brew 12 was carried out to illustrate the effect of an increased carbon dioxide flow of 12½ l/min. By mistake, the recirculation pump was not switched on, which is why the data is not used.

In the subsequent experiments, the carbon dioxide dosage was 12½ l/min in Brew 13 and 25 l/min in Brew 14 and 15, while other conditions are as in the experiment with Brew 11 with carbon dioxide dosage during the injection and 15 minutes of recirculation. In all these experiments, the size of the trub cone was the same - 3.5 cm - as at Brew 11.

Brew 13

FIGURE 4.11 shows the trub deposition for the experiment with Brew 13.
The figure shows that a firmly defined trub cone was formed in this experiment - the height was 3.5 cm. The low DMS values measured in the experiment are shown in TABLE 4.3. They show that the supply of carbon dioxide keeps the DMS values down, whereafter the expected increase is seen.

FIGURE 4.11. Trub deposition during trials with Brew 13.

TABLE 4.3. Data for measured DMS concentrations from experiments with Brew 13.

Sample	**DMS Brew 13**
Trial time	**ppb**
Start inlet	15
End inlet	15
Recirkulation	15
Start discharge	18
15 min discharge	20
30 min discharge	26
45 min discharge	34
End discharge	31

Based on the experiments, the following can be concluded:
- A reasonable trub cone cannot be deposited when carbon dioxide is supplied at the same time.
- A weakened but ok trub cone can be deposited when carbon dioxide is added during the inlet and the first 15 minutes of recirculation. This is largely independent of the added amount of carbon dioxide at the inlet.
- The optimal carbon dioxide dosage has not been determined.

We chose to carry out a final experiment with a boiling time of 30 minutes and a carbon dioxide dosage of 25 l/min, which is the maximum achievable with the carbon dioxide bottle used. This brew is compared to a normal pilsner brew cooked for 30 minutes.

During the later tests at Randers Bryghus and Humleland, we learned that there is no optimal carbonation dosage. It will depend on local conditions and the desired result: Is control of DMS in the whirlpool requested or is an additional effect on the taste and other properties of the beer requested.

4.3.3 Final trial with test whirlpool

The final experiment with the test whirlpool was carried out with Thy Pilsner. At Brew 14 Thy Pilsner was boiled for 30 minutes and this was compared to Brew 15 Thy Pilsner, also boiled for 30 minutes.

At Brew 14, a carbon dioxide dosage of 25 l/min was used during the injection and during re-circulation for the first 15 minutes. At Brew 15 no carbon dioxide stripping was used.

The results of the experiments appear in TABLE 4.4. FIGURE 4.12 and FIGURE 4.13 show the deposited trub in the two tests.

TABLE 4.4. Results from trials with Brew 14 and Brew 15.

	Brew 14	**Brew 14**	**Brew 15**	**Brew 15**
Trial time (min)	DMS ppb	Proces step	DMS ppb	Proces step
0	20	Start carbonation at inlet	22	Start inlet
15	13	Start recirc. with carbon dioxide	46	Start recirculation
30	14	Recirc without carbon dioxide	47	Recirculation.
45	21	Start discharge	52	Start discharge
60	31	Discharge	57	Discharge
75	33	Discharge	69	Discharge
90	33	Discharge	70	Discharge
Height, trub		3,5 cm	Height, trub	4,3 cm

For Brew 14, it can be seen that the injection of carbon dioxide causes a momentary drop in the DMS level from 20 ppb to 13-14 ppb. This level is maintained during the following 15 minutes of recirculation. In the last 15 minutes of recirculation, when the trub is deposited, the DMS level rises to 31 ppb, which is largely maintained throughout the discharge.

For the traditional Brew 15 it is seen, that the DMS level at the inlet is largely the same as in Brew 14, and that during the outlet it rises to just below 70 ppb.

In Brew 15, the highest value for DMS is 70 ppb at the end of the discharge. This value must be considered close to the value for total DMS. The Carlsberg measurement on the same samples indicates 48 and 60 ppb, which is in accordance with a large spread in all duplicate measurements.

Based on the final tests, it can be seen that:
- The method used for supplying carbon dioxide can halve the DMS content of the finished wort.
- 750 l of carbon dioxide has been consumed for 300 l of wort.
- The deposition of trub is slightly weakened but ok.

FIGURE 4.122. Deposited tube when experimenting with Brew 14 with carbonation – 30 min boiling.

FIGURE 4.13. Deposited trub when experimenting with Brew 15 without carbonation – 30 min boiling.

4.3.4 Experiment with high-gravity brewing with carbonated water

During the theoretical considerations in the project, it was discussed whether high-gravity brewing with carbonated cold water under pressure could be advantageous. The advantage should be that lowering the temperature to around 80°C will stop the conversion of precursors to DMS, and that the very small newly formed bubbles that arise from the pressure drop will form such a large surface that the carbon dioxide removes a reasonable proportion of DMS

.

We added just above 100 grams of carbon dioxide/hl of wort in 40 l of cold water in Cornelius barrels.

When pumping in, initially nothing happened. Suddenly the water boiled up due to the release of carbonation, which lasted a few minutes. The change in DMS was very limited and within the uncertainty of measurements. It is not considered that the method is applicable.

5. Testing and implementation of stripping at Randers Bryghus

Based on the experimental work with the test whirlpool, an experimental set-up was designed for full-scale trials with the process at Randers Bryghus. A well-functioning nozzle was designed for full-scale dosing of carbon dioxide and the possibility of dosing carbon dioxide just before the whirlpool as well as sampling from the whirlpool was established. The experimental work showed a halving of the DMS concentration in the whirlpool compared to operation without stripping. With a view to possible full-scale implementation, Randers Bryghus will test the method on a large type of beer over six months.

5.1 Adaptation and trials with pilsners

The Danish microbreweries are under severe pressure due to the corona epidemic, increasing raw material prices and fierce competition. In addition, Randers Bryghus has a large proportion of contract brews, with which experiments cannot be carried out, and the focus is on strong, complex brews. Experiments have been carried out with the 3 "pilsner character" brews that have been available.

5.1.1 Design of experimental setup

Hillerslev Maskinfabrik has constructed a nozzle system for use at Randers Bryghus and later at Humleland in Aarhus. As it has not been possible to connect a nozzle for the supply of carbon dioxide immediately before the inlet to the whirlpool, we chose to establish a nozzle system combined with sampling (before nozzles) in connection with the flow plate system between the wort kettle and the whirlpool. The developed nozzle worked excellently, and will be included in any future production.

Since there are no sampling valves on the side of the whirlpool at Randers Bryghus, Hillerslev Maskinfabrik has constructed equipment for taking samples from the man hole. The use of the equipment proved to be difficult due to a violent steam/carbon dioxide evaporation in connection with the pumping of wort to the whirlpool. Despite the difficulties, we consider that the results are satisfactory.

5.1.2 Testing with pilsner brew

Based on experiments 14 and 15 in the test whirlpool at Thisted, the first two pilsners brewed at Randers were brewed as experimental brews, and the third was chosen as a control.

The brews were double brews, which means that 10 hl of wort are boiled and processed at 1½ hour intervals and fermented together. The boiling time was 30 minutes. As with the experiments in Thisted, we added carbon dioxide before entering the whirlpool. Due to the relatively short time for isomerization of the hops, the hop dosage was adjusted.

Nozzle and sample outlet before the nozzle was established at the flow plate of the brewing plant, and a connection to the whirlpool was established with a hose of the same dimension as the piping.

At the first test brew, we established back pressure so that pumping over lasted 10 minutes. In the second test brew, there was no back pressure, and pumping over lasted 8 minutes.

25 l carbon dioxide/minute was added for the 8-10 minutes the pumping lasted. The whirlpool was managed as usual with a total process time of 1½ hours.
The indication of flow is uncertain.

TABLE 5.1 shows the measured values for the initial testing of the experimental design with the two brews. Data are shown for the measured values for DMS as well as for Total DMS – i.e. including precursors to DMS.

TABLE 5.1. Results from initial trials with Brew 1 and Brew 2, Randers Bryghus.

	Brew 1	**Brew 1**	**Brew 1**	**Brew 2**	**Brew 2**	**Brew 2**
Sample	Trial time min.	DMS ppb	Total DMS ppb	Trial time min.	DMS ppb	Total DMS ppb
Before nozzle	0	22	41	0	17	66
	5	27	112	4	24	60
	10	37	89	8	35	67
Before whirlpool	0	15	63	0	12	58
	5	17	71	4	16	74
	10	27	98	8	23	74
Whirlpool	5	12	-	4	9	72
	10	18	72	8	10	92

In Brew 1 it can be seen that the DMS level in the whirlpool is roughly 50% of the level in the wort that comes from the wort kettle.

In Brew 2 it can be seen that the DMS level in the whirlpool is roughly 40% of the level in the wort that comes from the wort kettle. No problems with trub settling was observed.

We choose to heat treat the samples converting precursors to DMS before analyzing for DMS. The variations of Total DMS in Brew 1 is explained in lack of experience.

Due to bad beer taste originating from the hose in Brew 1, the hose was changed between the two brews. The new the hose was longer than the one used in Brew 1. This is probably the reason for the greater effect of the carbonation in Brew 2.

5.1.3 Sensory evaluation of pilsner brew

The two test brews, together with a pilsner brew on tank, were evaluated at an expert tasting. The tasting showed, that Brew 1 was affected by a rubber/chemical taste from the hose. The author remembers the taste from a complaint on a large beer batch several years ago.

Brew 2 tasted better than the beer from tank. Although tank beer usually appears as the freshest, the difference was described as an increased freshness and friendliness in Brew 2. It was later described by the brewery as "a delightfully successful brew".
It is concluded that the method is suitable for Randers Bryghus for the production of pilsner/lager beer and similar types of beer.

5.2 Collection of knowledge about stripping with carbon dioxide

The experiments at Randers Bryghus, where we could not establish the injection of carbon dioxide immediately at the entrance of the wort line into the whirlpool, strengthened the assumption that the contact between carbon dioxide and the 100°C hot wort in a section of the wort line provides extremely effective removal of DMS. However, we cannot be completely sure, because the effect may also be due to an increased removal of DMS by the stripping that takes place in the whirlpool, where the carbon dioxide bubbles up through the wort that has been collected.

The experiments at Randers Bryghus also drew attention to a surprising improvement in the taste of pilsner beer in connection with the stripping. In agreement with the Danish Environmental Protection Agency, we have therefore carried out increased knowledge gathering primarily aimed at stripping warm wort with carbon dioxide.
The Scandinavian School of Brewing has contributed to this collection of knowledge with the report "Review and Literature about Wort Stripping", Appendix 5.
.
We have chosen to present two very different well-established systems, a German doctoral thesis and to highlight some interesting facts.

5.2.1 Meura Ecostripper

The system consists of a wort boiler, a whirlpool or settling tank, a column for wort stripping and a wort cooler. A traditional brewery can thus be expanded with the stripping system.

In the wort kettle, the wort is heated close to the boiling point, and it remains in the kettle for 30-50 minutes at 100°C with light stirring without significant evaporation. This provides sufficient isomerization of hops, formation of unwanted volatile components, destruction of enzymes as well as sterilization and formation of hot trub.

In the stripping column, unwanted volatile substances are stripped out by injecting clean steam in countercurrent with the hot wort that trickles downwards. This process is extremely efficient. In the whirlpool, either a normal process is carried out, or a Meura settling tank is used, which allows for the collection and recycling of wort in the hot trub, as this is carried out without contact with air.

Meura describes:
- An evaporation rate of 1-1.5%
- Improved foam stability
- Limited final darkening below 1 EBC
- Smaller amount of aldehydes

5.2.2 Steinecker Boreas wort stripping system

Below in FIGURE 5.1 is shown the Steinecker Boreas wort stripping system, where the wort is treated after the whirlpool, and where a stripping gas containing carbon dioxide, nitrogen and air (with oxygen) is used. The wort flows down the wall of the stripping vessel and evaporates unwanted substances into the stripping gas. A 50% reduction in evaporation is indicated, and the produced beer appears very similar to "originally" produced beer with a slightly better taste rating.

At first glance, the use of air and thus oxygen in connection with hot wort seems inappropriate, but the literature (Feilner et al, Berlin 2013) states that stripping can be done with air, carbon dioxide or nitrogen. The explanation is that water is constantly evaporating from the wort, and that this vapor forms a phase boundary that oxygen cannot pass through. Their measurements of color showed no difference.

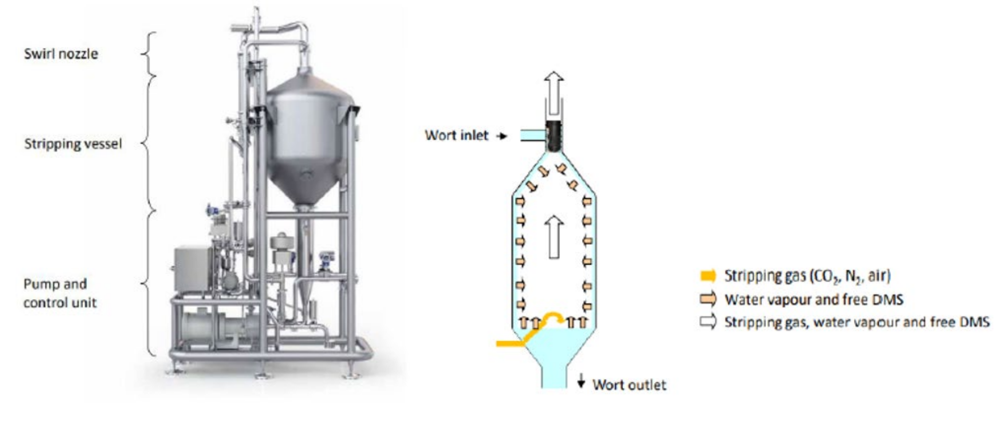

FIGURE 5.14. Outline of the Steinecker Boreas wort stripping system. Steinecker homepage – Boreas wort stripping.

5.2.3 Forced removal of undesirable aromas from beer wort

In connection with literature review, Dr. Ing. Roland Feilner's doctoral thesis "Forciertes Austreiben von unerwünschten Aromastoffen aus Bierwürze" is found particularly interesting and selected elements of the thesis are included below. In addition, chapter 9 Summary and chapter 10 Outlook of the thesis are attached as Appendix 6.

5.2.4 Literature summary

- The higher the temperature in the treated wort, the less amount of strip gas must be used.
- The color of the wort is not affected.
- ESR – Electron Spin Resonance (oxidative stability) is affected with slightly better durability
- Hop components are not affected.
- Since water molecules are very energetic near the boiling point, some water evaporates in connection with stripping.
- Steinecker indicates the possibility of lowering the level of aging carbonyls in finished beer to approx. 50% for 2-furfural, 90% for 2-methyl-butanal, 50% for 3-methyl-butanal and 80% for benzaldehyde.
- Meura indicates lowering of the amount of aldehydes.

5.2.5 Conclusions

- The reductions of DMS seen in the present project are consistent with reductions seen in the literature.
- The described reductions in the wort's boiling time agree well with the results of the present project.
- Reductions of unwanted aroma substances have not been comparable.
- The clear taste and aroma improvements registered in the present project (see also chapter 12 Humleland) do not correspond to the literature, which describes marginally improved taste.

- Preservation of the wort's color does not agree with the color change found in the present project. A little brighter and some light color was recorded. Darker color is traditionally associated with oxidation.
- Evaporation at Humleland was measured at 2.9% (see chapter 6), which is more than stated in the literature, but the stripping used was unnecessarily intensive, and about double the intensity at Randers. After correcting evaporation to less than 2%, there is agreement with the literature.

5.3 Construction and testing of experimental facilities at Randers Bryghus

The equipment for stripping with carbon dioxide is very simple, which is why experimental plants and commercial plants are the same. As an adaptation to the local conditions, we chose to test a representative selection of the many types of beer brewed at Randers Bryghus.

25 l of carbon dioxide per minute was supplied with the nozzle placed on the brewery's flow plate.
The focus of the project has been the control of DMS and thus shortening the boiling time. The many strong beer types that are brewed in Randers Bryghus mainly have a small content of pilsner malt and thus a small content of DMS. However, there are other flavoring components in beer that can influence the boiling time, which is why the experiments were organized to shed light on the possibilities of shortening the boiling time for different types of beer.

40 minutes of boiling time was chosen because the changes to the hop dosage are simple, and because we were sure that the treated beer would be sellable. The comments below on the various beers are primarily given by the brewery's co-owner and brewer Jonas Jacobsen. The present authors agree with the assessment.

5.3.1 Testing with different types of beer

We tested all the beers available for the project. It was not possible to carry out tests on salary brews.

Tulles Frokostøl 2,4 % vol.
The comments were primarily: "Light and fresh expression, which a lunch beer should have"
It was compared to a 1-year-old beer, to get a sense of the basic flavors in an untreated beer. It didn't really make sense.

Barely ½ year later, the beer is taste tested by Jonas Jacobsen and he states that it has maintained its quality.

Sekretæren, Belgisk Trippel 8,2 % vol.
The comments were: “Softer mouth feel. A bit like bottle aged beer. Approved”

Strandløven, Wittbier 5,0 % vol.
The test brew was compared to the same beer bottled 2 months earlier.
The comments were: Equally good beers, approved and fine. The treated test beer is slightly lighter and has a slightly lighter expression.
The older beer is a little more flavorful and the treated test beer a little easier to drink.

Nonnen, klassisk Belgisk Abbey ale uden krydderier 6,0 % vol.
The test brew was compared to the same beer bottled 3 months earlier
The comments were: Fresh and nice, better than the older beer.

Midnight Oil, Imperial Stout 10,9 % vol.
The test brew was compared to the same beer bottled a year earlier.

The comments were: OK beer, but outside the category. The older one is clearly better and really good.

5.4 Putting the stripping method at Randers Bryghus into operation

Randers Bryghus boils around 80 l of wort away for each brew of 10 hl, which gives an evaporation of 8%. If the boiling time is shortened by 30 minutes, above 3 m3 of natural gas is saved per brew (calculation according to Kunze, 2019). It is thus not the economics of the individual brew that will be the driving force behind the introduction of the stripping method.

More important is the 30 minutes saved for boiling, which also saves 30 minutes in the last brew of the day, as well as a less pressured production planning. The method will therefore make it possible to brew 3 brews in one day instead of the usual 2 brews. This will be economical, capacity-wise and environmentally advantageous.

Randers Bryghus will not change the traditional brewing methods without the changes being thoroughly researched and incorporated. Based on the positive test results, it has therefore been decided to test the strip method on a large type of beer for 6 months.

In addition to the shortening of long brewing days, the strip method is expected to produce only marginal changes in production.

Instructions will be verbal, as is customary at the brewery and in the industry.

Any problems with haziness will be handled with a slightly sharper filter in connection with the cooling down. Alternatively, centrifugation will be applied.

The focus will be on feedback from customers and beer tastings.

6. Supplementary testing at Humleland and general comments on quality

In addition to the test of the production method at Randers Bryghus, a test was also carried out at the Humleland brewery. Based on the testing and the project's results in general, Humleland will implement the method in the brewery's production. During the work with the brewerys it has become clear, that the treatment of the hot wort with carbon dioxide do have an effect on the beer quality. This effect is discussed.

6.1 Construction and testing

Humleland has a brewing unit that is the same size as the brewery at Randers Bryghus. The wort kettle and the whirlpool function are, however, integrated. We therefore chose to carry out the stripping in the pipeline used for recirculation. This means that the carbon dioxide was added to the nozzle directly before the inlet to the wort kettle/whirlpool, and that the added carbon dioxide bubbled up through the entire brew. This construction is very similar to the one used in the experiments in Thisted.

As with Randers Bryghus, planning was difficult. It was not possible to carry out trials in the run-up to Christmas, and later there were long intervals between suitable brews. In January 2024, we had the opportunity to compare two lager-type brews that had been brewed with an interval of two weeks. In order to even out the time difference in the brews, we chose to taste the beers in March 2024.

The wort was boiled for 40 minutes, and the same carbon dioxide flow was used as in the experiments in Randers. However, the supply was maintained for 15 minutes, which means that almost twice the amount of carbon dioxide was used per hl compared to experiment 2 in Randers. Boiling for 40 minutes is chosen because the changes in the hop dosage are therefore minimal.

6.2 Test results from Humleland

Results of DMS analyzes from the two test brews are shown in TABLE 6.1. The untreated brew shows DMS values of 17-28 ppb DMS. The treated brew shows values of 17-19 ppb DMS. The measured values are low because the brewhouse is relatively small and open, so DMS easily evaporates. The values are slightly lower in the treated brew, but they are all well below the taste threshold for DMS.
A water evaporation of 2.9% was measured.

When tasting, the treated brew appeared a bit lighter than the untreated one, and there was a small amount of sediment that had not settled. A light color of the beer is traditionally interpreted as the absence of oxidation.

TABLE 6.1. Results from trials with Brew 1 and Brew 2, Humleland.

	Brew 1	Without carbon dioxide		Brew 2	With carbon dioxide
Sample	Trial time min.	DMS ppb		Trial time min.	DMS ppb
Before nozzle	30	23		30	17
	60	17	Start carbon dioxide	40	11
	75	n.a.	End carbon dioxide	55	19
	90	28			

It is estimated that the haze is due to a reduced whirlpool efficiency, which can be handled with a filter.

There was a big difference in the taste and aroma qualities of the two brews, as the untreated beer appeared like an ok pilsner fermented with dry yeast, while the treated beer appeared fresh and friendly - clearly better as it was fermented with fresh propagated yeast. We were all surprised by the big difference.
The flavor profile was much the same as with trial brew 2 in Randers, just more distinct in terms of freshness.

The light color and flavor profile of the treated beer is consistent with the fact that almost double the amount of carbon dioxide per hl was used in relation to experimental brew 2 in Randers.

6.3 Resource consumption and commissioning

Humleland boils around 50 l away for each 10-hl brew, which gives an evaporation of 5%. Using 150 kWh for boiling for one hour, boiling for 40 minutes gives a saving of 50 kWh. It is thus not the economics of the individual brew that will be the driving force for putting the method into use.

At Humleland it has been decided that the method will be implemented. Especially because of the large difference in the taste of the two test brews, but also because of the savings - including the possibility of using the cheaper and easy-to-handle dry yeast rather than wet yeast to a greater extent.

The knowledge that the carbon dioxide stripping does not affect the hop flavor contributed to the decision. The impact on beer that is dry hopped with higher amounts of hops will be tested.

The implementation will take place in direct collaboration between owner and brewer. Instructions will be in verbal, as is customary in the industry.

Since the sale of Humleland's beer takes place in a large bar in the same room as the brewery, there is ample opportunity to obtain comments from the bar guests.

6.4 The quality of the beer – control of DMS

When you consult the specialist literature on wort boiling, the purpose is, among other things, described as a control of DMS, and the recommended boiling time of one hour or slightly longer is chosen to ensure boiling away of free DMS and DMS from precursors.

In the small breweries, DMS is not a problem, but they still boil the traditional hour.

Also, a barley variety is marketed that does not contain DMS or precursors to DMS. This has, however, not resulted in the use of malt from this barley with the aim of shortening the boiling time in the breweries.

The above can only be justified by the fact, that there are substances other than DMS that must be removed during boiling.

During the stripping at Humleland, as described in the above section, 2.9% was evaporated and the evaporated liquid was condensed. When assessing the taste and smell of the condensate, the description was: Strongly repulsive, astringent, chemical, and in every way unpleasant.
Different from the taste and smell that occurs in the first distillate in connection with the distillation of beer, and different from the smell that is registered during the fermentation of beer.

It is estimated that stripping has removed a significant amount of substances with an unpleasant taste and smell, which are otherwise not removed during the production of beer, and this contributes to the better sensory quality of the test brews, which we have recorded in connection with our work.

All breweries, and especially the large breweries, make great efforts to avoid oxidation of the hot wort, but all breweries that the present author is aware of pump the boiling wort into a whirlpool with a relatively large surface area and with an initial free fall. When stripping with carbon dioxide at the inlet at the whirlpool, the carbon dioxide will help to displace the air/oxygen, and it will in itself provide protection against oxidation.

It is estimated that for almost every single beer that a brewery produces, there is a technique and a level for adding carbonation to the hot wort, that gives an optimal effect on the taste.

The method is hardly applicable for very dark and strong beers.

7. Conclusions and perspective

It has been demonstrated that the brewing time of the wort in a traditionally built brewery with great probability can be reduced to 30 minutes for pilsner and similar types of beer with a large content of pilsner malt. It is further shown that the boiling time for most types of beer can very likely be reduced to 40 minutes.

It has further been demonstrated that the treatment technology used, stripping with carbon dioxide at the inlet to the whirlpool, will most likely result in the treated beer tasting almost like the same beer brewed traditionally, except that it will taste a bit fresher and better.

It is estimated that the method can be further developed so that the effect becomes stronger, whereby the boiling time can be further reduced.

It is estimated that the method can be constructed so that the degree of freshness in each individual brew can be chosen.

It is estimated that evaporation during the "boiling time" can be reduced to 2% or less.

It is estimated that by using the method in connection with brewing systems for ordinary atmospheric boiling, results related to quality and environment can be achieved on a level with - or better than - what is achieved in complex boiling systems.

Perhaps the method can become BAT technology.

8. References

Feilner Roland;"Forciertes Austreiben von unerwünschten Aromastoffen aus Bierwürze"; Technischen Universität Berlin; 2013.

Feilner et al; Technischen Universität Berlin; 2013.

Krottenthaler et al.; Optimizing brewhouse technology with reference to dimethyl sulfide; 43. Technologisches Seminar; EBC Brewing Science Group, Wolnzach; 2010.

Wolfgang Kunze; Technology Brewing and Malting, VLB Berlin; 2019.

Wilson & Booer; Control of the Dimethyl Sulphide Content of Beer by Regulation of the Copper Boil; J. Inst. Brew Vol. 85, pp 144-148; 1979.

Kotiaho,Tapio & Lauritsen, Frants R.; Membrane Inlet Mass Spectrometry; Comprehensive Analytical Chemistry XXXVII, 2002, Chapter 16, p. 531-557.

Steinecker homepage – Boreas wort stripping.

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Appendix 1. Review of Literature about DMS and Whirlpool

Appendix 2. Sensory Analysis

Training in tasting DMS was performed with reference samples from Aroxa (http://www.aroxa.com).
A group of 2 previously trained and 2 untrained judges was assembled, and a short intro with the topics "How we train", "What is DMS" and "Taste technique" was followed by the actual tasting.
The training was carried out on different substrates, as the aim was to use the sensory evaluation as the primary evaluation parameter throughout the project. Both wort, alcohol-free beer and light lager were therefore used, which were spiked with different concentrations of DMS ranging from 37.5 ppb to 150 ppb.
Guidance prepared by KU/Scandinavian Brewers Association was used, see appendix 2.1.
The training was carried out by a project participant with previous experience in sensory analysis and in training taste panels.
The training was conducted as an open tasting, where the individual judge on an unmarked line put X at the perceived intensity of the DMS taste in the sample. When finished, each individual's impressions were compiled on an overhead for an open discussion and eventual repeated tasting.

Low/none High/intensive

Unfortunately, the results showed that even at high concentrations (up to 150 ppb) a fairly common impression of taste and intensity could not be established. Only 1 of the 4 taste judges (brewer with many years of experience) showed an acceptable result when assessing the flavor intensity of DMS.
The conclusion was therefore, that sensory analysis cannot be used as the primary method for assessing DMS content in the phases of the project and at different stages in the brewing process. It was also concluded that DMS tasting in the context of this project is only to be used in the finished beer.

Appendix 2.1

Aroxa

http://www.aroxa.com

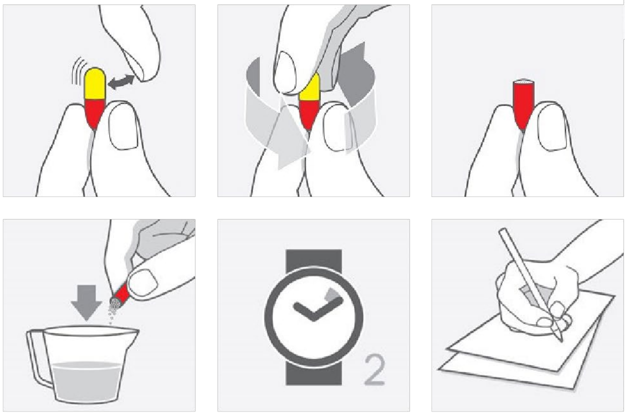
Flavour standards for:

- Beer
- Cider
- Soft drinks
- Water
- Wine
- > 70 different compounds

aroxa™

Beer Flavour Evaluation 1

Preparation of flavour samples



Beer Flavour Evaluation 2

1

Sensory Analysis Methodology

1. Swirl the glass at an arms length for a few seconds
2. Lead the glass towards your nose taking several small sniffs – note the sulphury flavours
3. Take one long sniff
4. Cover the glass with one hand and swirl to create headspace – remove hand and take one long sniff
5. Take a mouthful of beer, swirl in the mouth and swallow while breathing out through your nose

The retro-nasal perception can be improved by holding your nose while taking the mouthful of beer

Beer Flavour Evaluation 3

DMS = Dimethyl Sulphide

Associated terms:
- Sweetcorn, cooked vegetable, tomato sauce

Origin:
- Formed from pre-cursor in malt
- Should be removed during kilning and wort boiling
- Produced by wort bacteria
- Can be formed by yeast

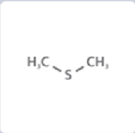
Significance:
- Normally off-flavour

Flavour compound:
- Dimethyl sulphide
- CH_3-S-CH_3
- 150 µg per capsule

Typical concentration in beer:
- 10 – 150 µg/l

Flavour threshold:
- ~ 25 µg/l

Flavour wheel No.:
- 0732



Beer Flavour Evaluation 4

Appendix 3. Analysis of dimethylsulfide in beer wort using Membrane Inlet Mass Spectrometry (MIMS)

A detailed description of the MIMS methodology can be found here:

Tapio Kotiaho and Frants R. Lauritsen, Membrane Inlet Mass Spectrometry. *Comprehensive Analytical Chemistry XXXVII*, 2002, Chapter 16, p. 531-557.

All analyses, both field analyses and frozen samples, are carried out by Professor Frants R. Lauritsen, University of Southern Denmark (SDU) or a trained laboratory technician under his supervision.

Analytical procedure.

For the analysis of the samples a membrane inlet of the sample cell type thermostated to 40 ºC is used and quantification is done using an internal standard (diethyl ether).

The analysis is carried out as follows (see Figure 1):

- At time zero, the sample cell is filled with clean water
- After 4 minutes, the water is replaced with the first sample
- After another 4 minutes, the internal standard diethyl ether (DEE) is injected into the sample cell achieving a final concentration of 23.6 ppb in the sample cell.
- After a further 4 minutes, the measuring cell is rinsed 3 times with clean water before the next sample is filled into the sample cell.

Quantification:

The DMS signal is monitored at m/z 62 (Figure 1, blue), while DEE is monitored at m/z 74 (Figure 1, red). The concentration of DMS in the sample is determined using the formula below for the use of internal standards (the principle is described in detail in Appendix A).

$$[DMS] = \frac{I_{DMS}}{I_{DEE}} \cdot 20\ (ppb)$$

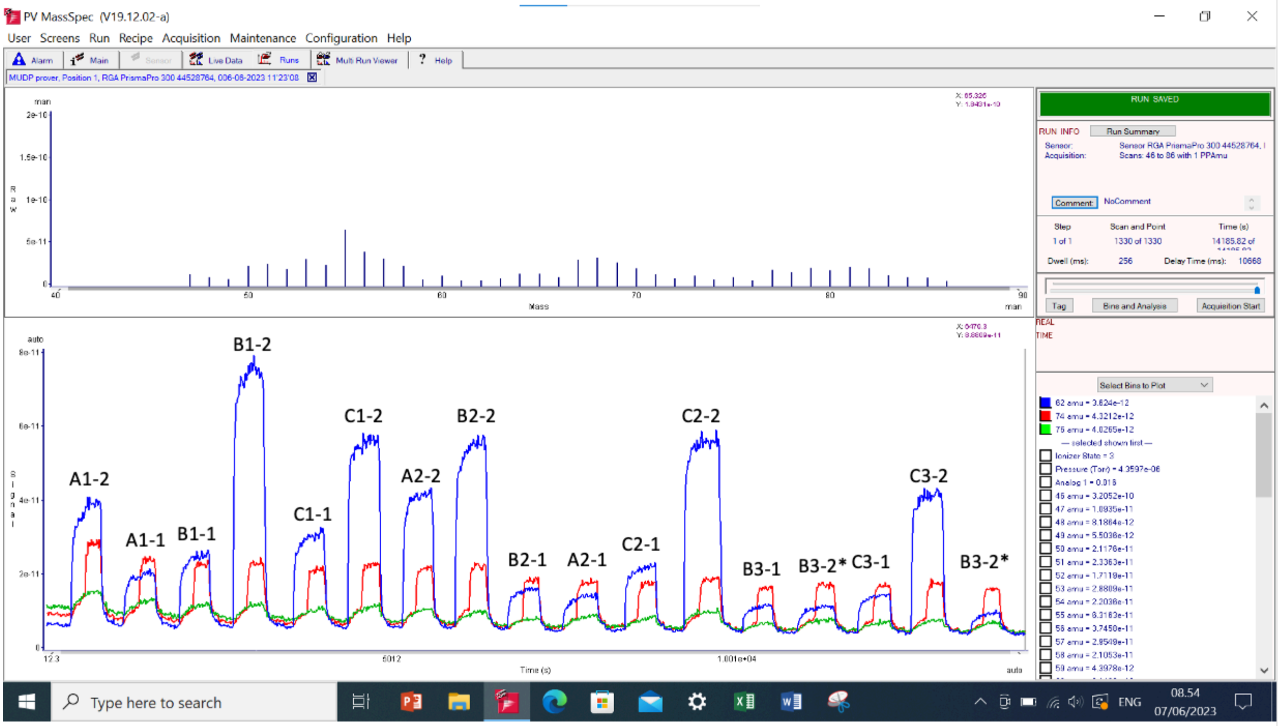
I_{DMS}: The measured intensity of the DMS signal
I_{DEE}: The measured intensity of the DEE signal

Before calculation a slowly increasing background signal measured at m/z 75 (Figure 1, green) must be subtracted from the two intensities "I". Reservations must be made for the calculations, due to uncertainty of the DMS content in the supplied DMS tablets used to produce the DMS standard. Further, an uncertainty of estimated 10% is caused since the exact background increase is unknown. This would require a blank herb sample (no DMS).

DEE is used as internal standard due to comparable physicochemical properties with DMS, especially comparable low boiling point (34.6 °C and 37.3 °C respectively).

Professor Frants R. Lauritsen, SDU (31-08-2024)

Analyse af DMS prøver fra Randers Bryghus den 02-06-2+23



Prøven B3-2 afveg fra de øvrige prøver ved at have et meget lavt væskeindhold (< 100 mL) i prøveflasken.

Figure 1. An example of the analytical procedure using a dataset from Randers Bryghus showing the analytical sequence for 17 samples. Blue is the DMS signal (m/z 62), red is the internal standard DEE (m/z 74 and green is the background (m/z 75).

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Appendix 3.1 Appendix A: Use of internal standard when analyzing DMS in beer wort

Analyte: Dimethylsulfide (DMS)
Characteristics: MW 62,1 g/mol bp 37,3 ºC density 840 g/l

Internal standard: Diethylether (DEE)
Characteristics: MW 74,1 g/mol bp 34,6 ºC density 713 g/l

Formula used:

Equation for standard addition $\frac{I_{DMS}}{[DMS]} = F\left(\frac{I_{DEE}}{[DEE]}\right)$

I_{DMS}: Measured background corrected signal from DMS at m/z 62
I_{DEE}: Measured background corrected signal from DEE at m/z 74
[DMS]: Concentration of DMS in the sample solution
[DEE]: Concentration of DEE in the sample solution
F: Sensitivity factor

Production of standards:

DMS: Dissolve 150 µg DMS from the tablet in 20 mL water ⇒ 7,5 ppm
100 µl solution is added to 30 mL of water in the analysis cell ⇒ 25 ppb

DEE: Dissolve 0,2 µl of pure DEE in 20 mL of water ⇒ 10 ul/l
With a density of 713 g/l, the mass/volume is calculated to be 7.1 ppm
100 µl solution is added to 30 mL of water in the analysis cell ⇒ 23,7 ppb

Determination of sensitivity factor:

Concentrations used: [DMS] = 25 ppb; [DEE] = 23,7 ppb
Measured values (relative) I_{DMS} = 100 (A); $I_{DEÆ}$ = 90 (A)

Determination of F: $F = \frac{I_{DMS} \cdot [DEE]}{[DMS] \cdot I_{DEE}} = \frac{100 \cdot 25\ (ppb)}{90 \cdot 23{,}7\ (ppb)} = 1{,}17$

Determination of DMS concentration:

$$[DMS] = \frac{I_{DMS}}{I_{DEE}} \cdot \frac{[DEE]}{F} = \frac{I_{DMS}}{I_{DEE}} \cdot \frac{23{,}7\ (ppb)}{1{,}17} = \frac{I_{DMS}}{I_{DEE}} \cdot 20\ (ppb)$$

Appendix 4. Litterature review – DMS formation

Appendix 5. Review of Litterature about Wort Stripping

Appendix 6. Dr. Roland Feilner: Forciertes Austreiben von unerwünschten Aromastoffen aus Bierwürze, Zusammenfassung und Ausblick

Climate friendly wort management in breweries

Projektet vedrører forkortelse af kogetiden ved ølbrygning. Dette opnås ved stripning af den varme urt med kulsyre.
Umiddelbart inden den kogende urt løber ind i whirlpoolen sprøjtes kulsyre ind i urten via en dyse.
Kulsyreboblerne danner en overflade til urten, hvorigennem Dimethylsulfid (DMS) og andre dårligt smagende stoffer fordamper og fjernes med kulsyren.
Da urtens kogetid er betinget af at DMS mm. skal koges bort, kan kogetiden for lyse og lette øltyper forkortes til omkring en ½ time.
Analyser, foretaget af SDU, blev gennemført med MIMS – Membran Inlet Mass Spectrometry – hvor prøven diffunderer gennem en membran, og sorteres efter masse.
En testwhirlpool på 400 liter blev opstillet hos Thisted Bryghus, hvis bryg er en faktor 10 større end bryggene hos Randers Bryghus.
Målinger af DMS angav en halvering af DMS-indholdet i den kulsyrebehandlede urt.
Forsøgene viste, at en fornuftig trubkegle kunne etableres i whirlpoolen ved samtidig indblæsning af kulsyre. Sammenlignende forsøg med øl af pilsnertype hos både Randers Bryghus og hos Humleland viste, at det behandlede øl blev bedømt ”friskere” og ”klart friskere, lysere og bedre”.

Det er dokumenteret, at kogetiden ved urtkogning af pilsner og lignende øltyper kan nedbringes til ½ time. Det er sandsynliggjort, at den anvendte metode giver den fremstillede øl en friskere aroma og smag. Dette uden at humlearoma og øvrige smagsmæssige parametre påvirkes.
Det er dokumenteret, at kogetiden for de fleste øvrige øltyper kan nedbringes til 40 minutter.
Der er udviklet en hurtigmetode til analyse af DMS.

The Danish Environmental
Protection Agency
Tolderlundsvej 5
DK - 5000 Odense C

www.mst.dk