

# Analyses of N-Nitrosodimethylamine (NDMA) in Malt and Beer

News from VLB Research Institute for Brewing and Malting Technology

Provided by  
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Malt and malt beverages generally still have a certain content of NDMA. Due to the high carcinogenic potential of this substance, continuous analyses especially of the different malt batches are essential for food safety. Therefore VLB Research Institute for Brewing and Malting Technology (head: Prof. Dr. Frank-Jürgen Methner) has established a reliable method for the analyses of NDMA in malt and beer. This method is part of the accredited scope of services of the VLB Central Laboratory.

NDMA is present in a variety of foods, such as some varieties of cheese, bacon, various cured meats, fish, canned fruit, and also in tobacco products. The presence of NDMA in beer was first reported at the end of the Seventies. Malt was found to be the main source of this contamination. The NDMA was formed during the drying process, where the malt was in direct contact with the hot flue gases used for drying. So, to avoid this contact, in 1981 the drying technique was generally changed into an indirect one. This led to a significant decrease of the level of NDMA in malt and thus in beer, where the NDMA is finally found due to its good water solubility. The formation of NDMA during the drying process is well researched. Mainly horde-nine, a secondary metabolite of the amino acid tyrosine, is formed in larger amounts

in the rootlets during the germination process of barley and reacts with the nitrogen oxides (NOx) of the flue gases to give nitrosohordenine in the first step. At temperatures higher than 60 °C, dimethylamine is generated from this, which then reacts with further NOx to give NDMA. Due to this, nearly 80 % of the NDMA is found on the surface of the grain. Thus a correct sampling of the malt is absolutely necessary prior to analysis, the sample must be representative for the whole batch. In order to prevent further reaction with NOx in the air, the samples have to be provided in air tight containers.

### Human exposure to NDMA has to be minimized

It is estimated that air, diet and smoking contribute to potential human exposure at levels of a few micrograms per day. Beer, especially when brewed from dark malt, is considered to be one of the main sources for the uptake of nitrosamines. As a result of the high carcinogenic potential of NDMA, exposure should be reduced to the lowest extent possible. Therefore in Germany there exists a technical reference concentration



Fig. 1: Capillary gas chromatography coupled with a new ANTEK 7090N Nitrogen detector for analyses of NDMA

of 2.5 ppb for malt and of 0.5 ppb for beer. The results of larger sample checks regularly performed by the state offices and even our own results show that some of the samples do not comply with these legal requirements. So, even over 25 years after the first discovery of NDMA in beer, the problem has not yet been overcome and there is a further need for regular investigations.

### VLB analytical services

The VLB Central Laboratory is accredited according to DIN EN ISO/IEC 17025:2000 and is the breweries' partner for chemical analyses of

- Hops and hop extracts
- Brewer's wort
- Beer
- Filter aids
- Spirits
- Water and waste water
- Special analyses

State-of-the-art laboratory equipment guarantees the execution of all orders in accordance with the regulations of the standard literature of analysis. Our laboratories are equipped with gas- and liquid chromatographs, mass spectrometers, atomic absorption spectrometers, image analysis devices etc.

All analyses are performed by qualified personnel only. Depending on the customers'

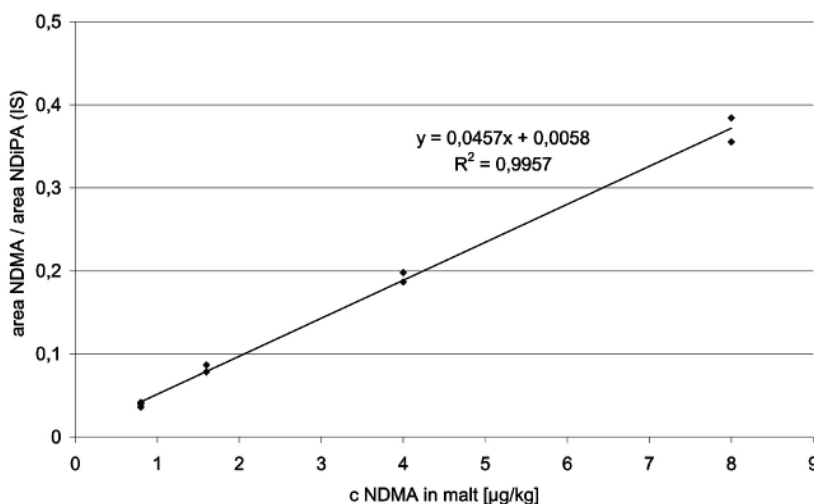


Fig. 2: Calibration plot for NDMA in malt based on four pairs of variates in the range of 0.8-8 µg/kg (ppb)

specifications, experienced chemists, food chemists, engineers, agronomists and biologists are at your disposal. Our well-trained staff is the basis for our successful performance at the highest level of quality.

#### NDMA detection by a modified MEBAK method

The determination of NDMA in malt and beer is performed based on the method of MEBAK but slightly modified. For malt analysis, this is extracted by application of a modified congress mash using 100 g of grist. Beer samples or the prepared modified congress worts are stabilized with sodium hydroxide solution in order to avoid the formation of artefacts during the following extraction step. For clean-up the samples are applied to an Extrelut® column using N-nitrosodisopropylamine (NDiPA) as an internal standard. The extraction is performed with dichloromethane. The extracts are evaporated at room temperature to a volume of approximately 1 mL and then are further concentrated using a slight stream of nitrogen gas to

blow off the liquid to exactly 0.4 mL. The analysis is performed by capillary gas chromatography coupled with a chemiluminescence detector. Therefore the GC is equipped with the new ANTEK 7090N Nitrogen detector (Fig. 1). Instead of MEBAK, a calibration based on four points of variates is used in order to achieve more reliable results in the low concentration range (Fig. 2). The limit of quantitation is 0.8 ppb for malt and 0.2 ppb for beer with a mean recovery of 90-110 % at this level and a precision of  $\pm 5$  %. The method has been developed by VLB's Research Institute for Brewing and Malting Technology and is part of the accredited scope of services of the VLB Central Laboratory.

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## +++ VLB Ticker +++

### ▶ 93 International Brewing and Engineering Congress

International meeting for the brewing, beverage and supply industry, 6 to 8 March 2006 in Regensburg, Germany (languages German/English)

### ▶ Craft Brewers Conference /BrewExpo, Seattle

Meet us at our stand at the BrewExpo America during the Craft Brewers Conference, April 11 -14, in Seattle, USA

### ▶ Excellence in Brewing Technology, Course 2006

Advanced 3-week training course for professional brewers, 6 to 24 November 2006, in Berlin/Germany

### ▶ 93 International October Conference

International conference for the brewing, malting and supply industry, 9 to 11 October 2006 in Berlin, Germany (languages German/English)

### ▶ Certified Brewmaster Course 2007

Comprehensive training course for prospective brewing professionals in Berlin / Germany, January to June 2007 in Berlin (languages German/English)

[www.vlb-berlin.org/english](http://www.vlb-berlin.org/english)

# 93. International Brewing and Engineering Congress

6 to 8 March 2006

Regensburg, Germany



International Congress for the  
Brewing- and Beverage Industry

Congress languages: German and English

Major topics:

- ▶ Filling of Beer: Innovation in Technology and Potentials for Optimisation
- ▶ Beverage Packaging: Technological and Legal Aspects
- ▶ Hops – Raw Material with Potential
- ▶ New Developments in Analysis
- ▶ Innovative Beverages

With contributions from

AC Nielsen, Ball Packaging, Baltika Brewery, Bavarian Research Institute for Agriculture (LRL), Bischofshof Brewery, Bitburger Brewery, German Brewers Association, German Cancer Research Center (dkfz), Hopsteiner, KHS, Kronen, NATECO, Simonazzi, Symrise, Technische Universität Berlin, Technische Universität München, University of Applied Science Lippe and Höxter, VLB Berlin, WIMG

Supported by



Registration and Information

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