



# NDMA in Malt and Beer



## NDMA (n-nitrosodimethylamine) in Malt/Beer

During the malting process, barley is forced to germinate, after which the grain is dried in kilns. This process freezes the sugar and flavour compounds for the brewing process. During the kiln drying process, nitrosamines may be formed in the grain, which could remain within the extract and still be present in the final brewed product. Many recently used techniques, minimise the formation of nitrosamines., However, low levels of these carcinogenic compounds still remain. Therefore, all malt used in the brewing process needs to be analysed for its nitrosamine content. It is important to monitor the final product as well as the malt, to help regulate the exposure of nitrosamines from the consumed liquid.

The Ellutia 200 Series Gas Chromatograph with an EL-WAX column was utilised alongside the 800 series Thermal Energy Analyser (TEA)

### GC Conditions

Injector Temperature	250°C
Liner Type	Focus Liner with Wool
Carrier Gas Type	Helium
Carrier Gas Control Method	Constant Flow
Splitless Time	0.8 min
Column Flow	1.0 ml min <sup>-1</sup>
Injection Volume	1 µl
Column	EL WAX 30 m x 0.25 mm x 0.25 µm
<b>Column Temperature Program</b>	
Initial Temperature	45°C/Hold 1 min
Temperature Ramp 1	20°C min
Column Temperature 1	130°C/0 min Hold Time
Temperature Ramp 2	200°C (10°C min <sup>-1</sup> )
Column Temperature	230°C/1 min Hold Time

### TEA Conditions

Pyrolyser Temp	500°C
Interface Temp	250°C
Sensitivity	250
Pump Type	Edwards nXDS10i



## Malt

### Sample preparation (Malt) –

Samples were extracted in duplicate. Each replicate had 50 grams of malt ground up and had 100 ml deionised water added. The extract was filtered through a Whatman Grade 1 filter paper and 1 ml of 10 ppm NDPA (n-nitroso di propylamine) internal standard was added to one extract (this generates a 100 ppb NDPA spiked sample). The samples were then made up to 100 ml with deionized water volumetrically.

To a vial, 10 ml of extract, 3 grams of sodium chloride and 10 ml of dichloromethane (DCM) was added and shaken for 5 minutes. Then the layers were left to separate for 15 minutes. The lower layer containing DCM was pipetted out into a clean vessel. 10 ml of DCM was added to the extract and the liquid/liquid extraction step was repeated. After this step, the DCM (final volume approx. 20 ml) was dried using 1 gram of sodium sulphate and then pre-concentrated to 1 ml under a nitrogen flow of approx. 1 l min<sup>-1</sup>. A 1 µl injection of the concentrated DCM was directly analysed.

The identity of the internal standard was confirmed against the 8 component nitrosamine mix standard. This shows that the NDPA internal standard used has the same retention time as the NDPA contained within the standard (figure 1).



The peak areas for the spiked sample compared with the standard for NDPA showed good correlation, indicating a good recovery of the internal standard, and thusly, indicating very limited losses of any potential nitrosamines from within the sample during the preparation steps. The unspiked sample showed no peaks within the retention times of any of the nitrosamines in this standard mix. As such, this malt sample showed no observable response for NDMA or any other nitrosamines.

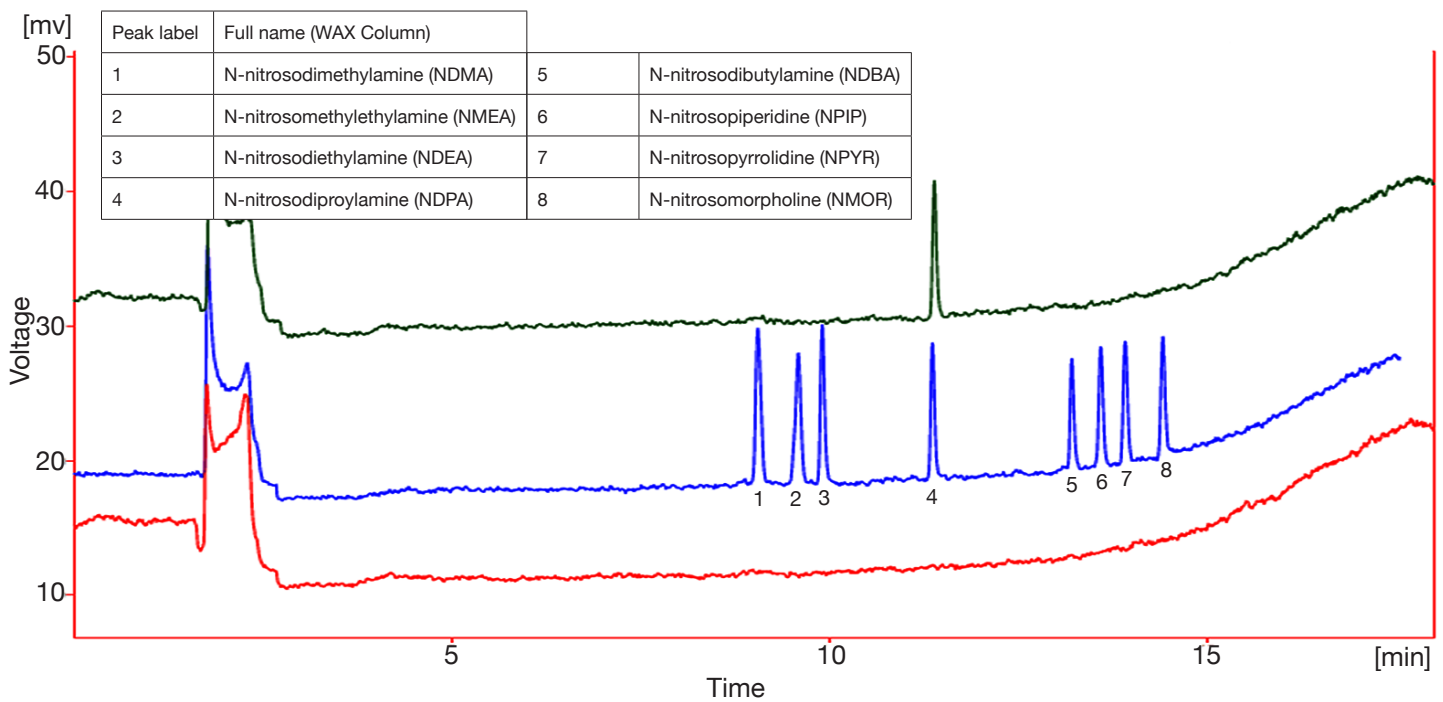


Figure 1. Blue - 100 ppb 8 component mix, Green – 100 ppb NDPA (internal standard) spiked malt sample and Red - unspiked malt sample.

Beer

25 ml of lager was sonicated for 10 minutes to remove any dissolved gases. 10 ml was transferred to a centrifugal tube, 3 grams of sodium chloride was added, followed by 1 mL of DCM and the solution was shaken for 5 minutes. The sample was then left to separate out for 15 minutes. The DCM layer was pipetted into a new vial and 1 gram of sodium sulphate was added to capture any remaining aqueous solution. A 1 µl injection of the DCM solution was directly analysed.

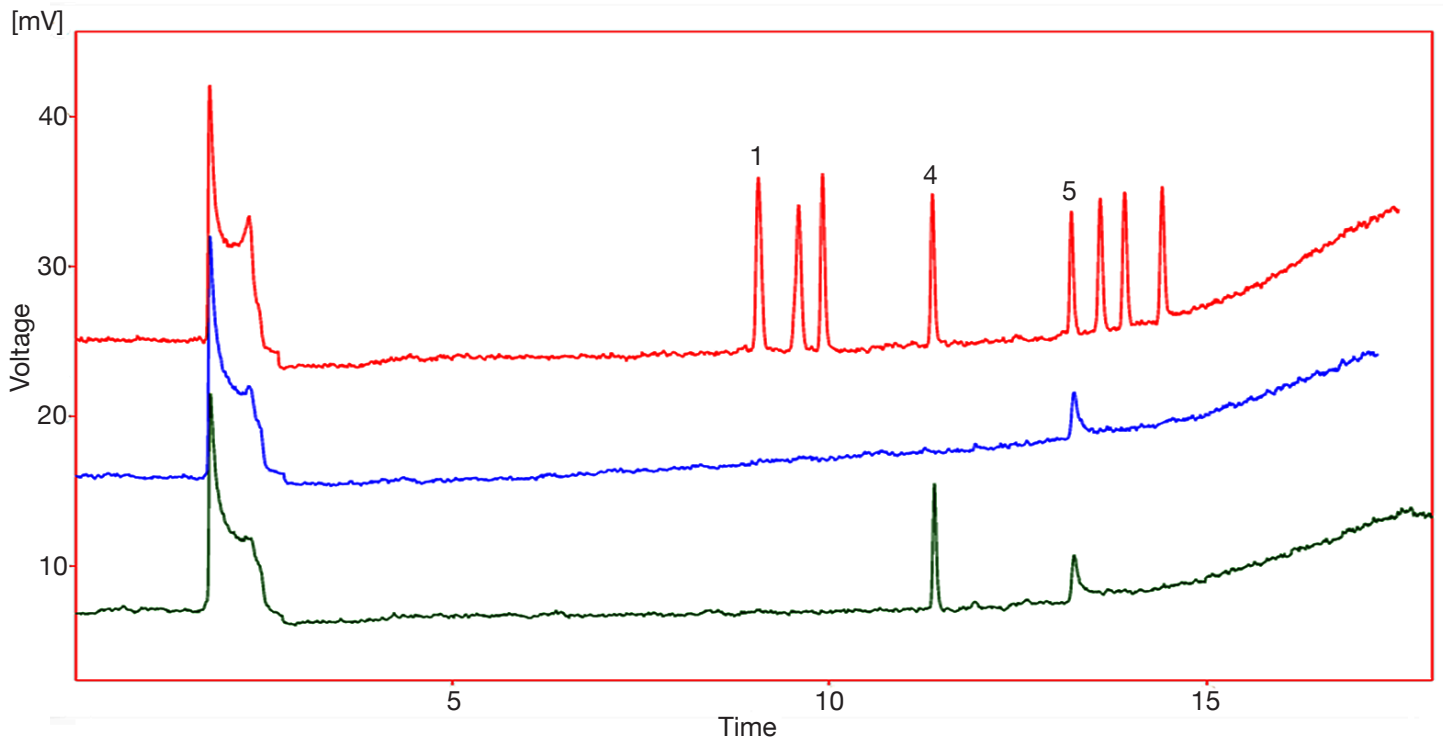


Figure 2. Red - 100 ppb 8 component mix standard, Green – 100 ppb NDPA (internal standard) spiked lager sample and Blue - unspiked lager sample. Peak 1 – NDMA, Peak 4 – NDPA and Peak 5 – NDBA.

NDMA detection limits for this instrument have recently been generated and show that <1 ppb NDMA can be seen. For the malt sample the results would be declared as none detected <1 ppb, the beer would also be declared as <1 ppb NDMA, however, there was a response found for NDMA (n-nitrosobutylamine) of 67 ppb.

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## Ordering guide

### TEA

TEA 810 - 230V	(Part no. 32000820)
TEA Pyrolyser Interface Kit	(Part no. 32020300)
NXDSi 10 Dry Pump	(Part no. 32020200)
Ozone Destroyer	(Part no. 32001060)

### Gas Chromatograph

200 Series Gas Chromatograph (No Detector)	(Part no. 20500430)
Elution Software, Single Instrument	(Part no. 23001001)
Colibrick - 1 Channel	(Part no. 23001022)
EL WAX 30 m x 0.25 mm x 0.25 µm	(Part no. 51100767)
GC Mounting kit (for EL Autosamplers)	(Part no. 30500018)
Ellutia Autosampler Control Software	(Part no. 23001012)

### Ellutia Liquid Autosampler offerings

Ellutia EL3100A Automatic Liquid Autosampler - 15 position	(Part no. 30500011)
Ellutia EL3000A Automatic Liquid Sampler - 121 position	(Part no. 30500010)
Ellutia EL3200A Automatic Liquid Sampler - 209 position	(Part no. 30500012)

### Accessories

2 ml Vials	(Part no. 20511101)
2 ml Vials Screw Caps	(Part no. 20511107)
1 µl Liquid Syringe	(Part no. 20511204)
Septa (pk50)	(Part no. 20512101)