

# Using automated sorptive extraction to detect trace-level odour-active compounds in plant-based foods

Detecting and eliminating off-odours in food is vital in the development of new products, but many of the most aroma-active compounds can be present at levels that make it difficult to implement widely-used analytical techniques such as purge-and-trap. We talk to food scientist Diana Owsienko, who has turned to Markes' HiSorb and Centri technologies to extract key analytes more quickly from smaller amounts of sample. As a result, it's now easier to run sensory and instrumental analyses on the same batch, for better correlation of results, all within a more streamlined, automated workflow.

## Understanding the aroma of food

Consumer satisfaction with the food we eat depends on a variety of sensory characteristics, of which aroma makes a vital contribution. But the sensitivity of the human nose to individual compounds also makes understanding the origin of certain aromas a uniquely tough analytical challenge.

One person who's acutely aware of this is Diana Owsienko, who works within the Aroma Chemistry Group at RISE, Sweden's government-funded network of research centres. The group is currently working on several collaborative research projects that involve developing new plant-based foods that are tasty, nutritious, and have an improved sustainability profile.

She explains the challenge they were facing: "One of our projects involves exploring alternative protein sources such as legumes. But legumes can produce a 'beany' odour, which can be undesirable if it's apparent in the final food product – so we were working to understand the compounds that give rise to it, and how to reduce their levels".

## Overcoming the limitations of existing methods

To investigate the causes of this off-odour, Owsienko and colleagues had been using purge-and-trap sampling. Gas-phase volatiles in the headspace above an aqueous protein-powder suspension were being collected on sorbent tubes, followed by thermal desorption and detection by GC-olfactometry and GC-MS. This combination of sensorial and instrumental detection techniques is commonplace in the field of food analysis, because it allows odours detected by the human nose to be matched to the chemical that gives rise to them.

“ The headspace and immersive sampling options on Centri help us reveal a broader spectrum of volatile compounds ”



### CUSTOMER

Aroma Chemistry Group,  
Research Institutes of  
Sweden (RISE)

### APPLICATION

Aroma profiling of legume-derived protein-rich powders

### CHALLENGE

Reducing the sample size needed to detect trace-level off-odours

### SOLUTION

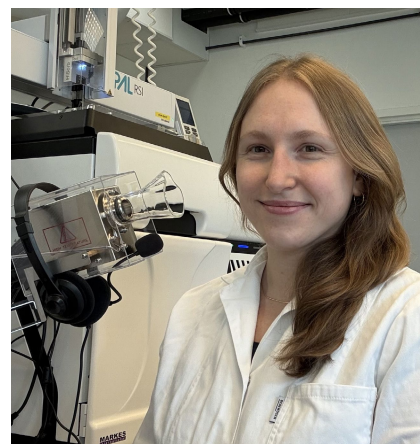
HiSorb™ high-capacity sorptive extraction, automated on the Centri® sample preparation and preconcentration platform

### RESULTS

Over 10-fold reduction in sample size, making it easier to carry out sensory evaluations and instrumental analysis on the same batch

But there was a problem, says Owsienko. They found that some of the compounds responsible for this beany off-odour were present at trace levels, which meant that they were working at the limits of their purge-and-trap method. One consequence of this, she explains, was that they needed to increase the sampling time, in order to maximise the amount of volatiles extracted.

However, this was unsatisfactory on two levels. Firstly, she says, it was a very lengthy workflow, with the sampling time of up to 3 hours exacerbated by the manual work involved in preparing, starting and terminating the sample extraction, as well as the need to clean the sampling bottles afterwards. “It was taking up to a full day to perform the purge-and-trap method to extract the aroma compounds from several samples in replicate”, she says. The second problem was that the large amounts of sample needed were hampering their ability to carry out sensory evaluations on the same batch of material within a reasonable timeframe.



Diana Owsienko using Centri in combination with GC-olfactometry.

At this point the team realised that they needed a more sensitive sampling approach. Following a technical discussion with Markes, it was clear that the Centri sample preparation and preconcentration system would be ideal for the purpose. As well as allowing desorption of sorbent tubes, Centri offers options for headspace-trap, SPME-trap, and (crucially for the RISE team) HiSorb sorptive extraction.

### Enhancing sensitivity with HiSorb and Centri

HiSorb involves sampling volatiles onto a probe, followed by desorption and preconcentration on a trap before analysis by GC. A key feature of HiSorb is the relatively large volume of sorptive phase, and Owsienko has found that this makes it much easier to achieve the desired sensitivity for the team's target analytes with a limited amount of sample. “HiSorb is great at extracting trace aroma compounds, meaning we need much less of the aqueous suspension: just 10–20 g, compared to 500 g for purge-and-trap”, she says. “This gives us much more flexibility during method development, because the amount of sample available is usually the limiting factor when we're optimising variables such as extraction time”.

“**HiSorb is great at extracting trace aroma compounds, meaning we need much less sample**”

She adds that a further sensitivity-enhancing benefit available on Centri was the ability to ‘enrich’ the sample, by extracting several samples (including those taken with different HiSorb phase combinations) and collecting the volatiles onto the same focusing trap prior to desorption. This resulted in the ability to discover trace-level aroma compounds that might otherwise have been missed, she says.

Another approach used by the team, Owsienko explains, was to use the system's capability for splitless desorption: “Low odour thresholds for trace compounds are a common problem in aroma analysis, so running our thermal desorption methods splitless means that we maximise the sensitivity for the injection”.

### Wide-ranging automation for time-efficient workflows

Automation of HiSorb on Centri also solved the workflow bottleneck. “It's much more convenient”, says Owsienko. “Because Centri does all the extraction automatically, all I need to do is weigh out the powder sample, add some water and maybe an internal standard, and then let Centri do the rest. That means I can use my time more efficiently, focusing on other things like data evaluation”.

And, she points out, automation of the entire sampling, extraction and analysis workflow means it's much easier to repeat the process. So if something goes wrong, or replicates need to be run, or a quick GC-olfactometry screen needs to be carried out, that's no trouble: “It's much more flexible”, she concludes.

## The vital role of correlating analytical and sensory data

In fact, that flexibility is a feature of the entire workflow, says Owsienko, with two aspects being the ability to use different sorptive phases and different sampling modes: “It’s great to be able to switch so easily between the different HiSorb phase combinations, and also between headspace and immersive sampling. Not only can this reveal a broader spectrum of volatile compounds, but it enables you to test which sampling method is better, without having to worry about the amount of sample you’re using”.

“ **The automated extraction available on Centri allows me to use my time more efficiently** ”

And being economical with the sample means that there is plenty available for sensory panel studies, she explains: “We can see which attributes the sensory panel is rating as high intensity, and that gives us useful information for the analytical side. So, for example, if the ‘beany’ attributes are flagged up as off-odours in a particular sample, then we know to focus on the corresponding compounds when we come to the GC–MS and GC–olfactometry work”.

Combined with the enhanced sensitivity, she says, this has enabled them to uncover trace-level off-odour compounds in legume-based protein, as well as new desirable compounds derived during treatment processes. This way, she concludes, they can evaluate the effect of different treatments on the aroma profile, and, in collaboration with other groups at RISE, also assess the impact on the functional properties of the protein, thereby developing products in which the problematic off-odours are eliminated.

She concludes with her thoughts on the impact this will have: “Through our research, and thanks to the role of Markes’ equipment in improving our workflow, we hope to improve consumer acceptance of plant-based food products. That in turn will contribute to one of the key aims of RISE – more sustainable food production and consumption”.



**Diana Owsienko** studied food chemistry and carried out her Ph.D. at Friedrich-Alexander-Universität Erlangen-Nürnberg, Germany, where she investigated the chemical composition of body odours, with a focus on age-related changes. Since May 2023 she has been working as a Researcher for RISE Sweden, within the Department of Food Research and Innovation, based in Gothenburg. There, she is part of the Aroma Chemistry Group, supporting customers with aroma-related questions, including identification of off-odours, as well as working on research projects dealing with plant-based food.

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