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Vacuum and Headspace – An Efficient and Fast Combination for the Extraction of Volatile Compounds

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Different methods of extraction of volatile compounds from the headspace are known. Most of these techniques are carried out at a pressure higher than atmospheric pressure due to the heating of the sample in a closed space. For these techniques, the sample has to be heated in order to quickly reach an equilibrium state in the headspace. Reduced pressure extraction techniques have also been shown to extract compounds over a wide molecular weight range.

The Flavour Research Laboratory of the Swiss Federal Competence Centre for Agricultural Research, Agroscope, has developed an innovative and cost-effective method for the extraction of volatile compounds for gas chromatography (GC) analysis under the name of Dynamic Headspace Vacuum Transfer In Trap Extraction (DHS-VTT).[1,2] The technique also limits artefact formation during the extraction process thanks to reduced temperature and extraction time.

The aim is to combine Headspace In-tube Extraction (ITEX) with a vacuum pump to obtain a dynamic extraction at reduced pressure.

Volatile compounds from the sample are trapped in an ITEX-filled needle using a vacuum of around 5–10 mbar. The extraction time depends on the compounds to be extracted but varies between 5 and 30 minutes. A temperature-programmed injector (PTV) completes the set-up perfectly to optimise the separation of volatile compounds in the GC-MS.

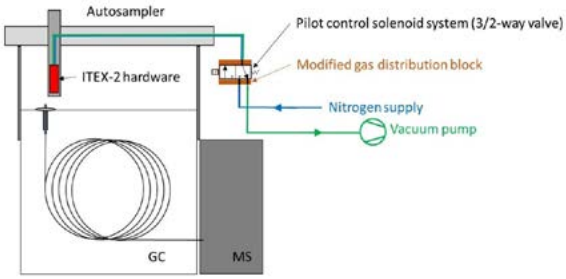

Fig. 1 Diagram of the GC–MS instrument with the autosampler and ITEX-2 hardware. Connection of the original nitrogen line to the new distribution block (in orange) and the solenoid valve. The vacuum and nitrogen lines are coloured green and blue, respectively.

The DHS-VTT technique significantly improves the extraction of volatile compounds from a complex matrix such as fermented dairy products in comparison with the ITEX and solid phase microextraction (SPME) method. The modification of the sampler is fast, economical and allows the use of commercial ITEX equipment. The method allows rapid extraction of target compounds using vacuum with little artefact formation or sample degradation. In addition, it is possible to extract large quantities of samples without having to replace the extraction equipment due to premature wear of the extraction parts or polymer. The results showed that the extraction equipment can be used for more than 850 injections without being modified; their relative standard deviation (total peak areas of 43 target volatile compounds over two weeks) was 9.6%. The technology is commercially available from CTC Analytics AG (www.ctc.ch).

DHS-VTT has many applications, such as working on projects requiring a large number of samples for metabolomic analysis.[3] The high extraction capacity of the ITEX needle polymer allows olfactometric analysis to be carried out with several panelists simultaneously without the limitations of the extraction media.

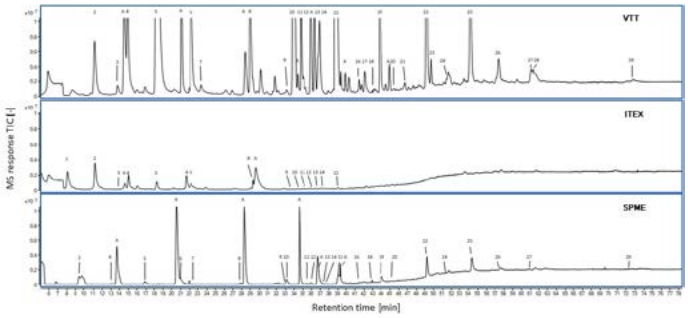

Fig. 2 Chromatograms corresponding to the volatile fraction of plain yoghurt extracted by DHS-VTT, HS-ITEX, and HS-SPME methods. Analytes from left to right: acetaldehyde, acetone, ethylacetate, butan-2-one, butane-2,3-dione, pentane-2,3-dione, hexanal, heptan-2-one, octanal, 3-hydroxy-butan-2-one, 2-methylpentan-3-ol, 2-hydroxy-3-pentanone, nonan-2-one, nonanal, acetic acid, propanoic acid, 2-methylpropanoic acid, undecan-2-one, butanoic acid, 2-phenylacetaldehyde, pentanoic acid, hexanoic acid, 6,10-dimethylundeca-5,9-dien-2-one, 2-phenylethanol, octanoic acid, nonanoic acid, δ–decalactone, decanoic acid, δ-dodecalactone. A = artifact.

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[3] P. Fuchsmann, M. Tena Stern, L. H. Münger, G. Pimentel, K. J. Burton, N. Vionnet, G. Vergères, *J. Proteome Res.* **2020**, *19*, 4019, https://doi.org/10.1021/acs.jproteome.0c00324.

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