COMBINING ION MOBILITY SPECTROMETRY WITH LATEST THERMAL DESORPTION TECHNOLOGY

GC-IMS ITEX DHS TECHNOLOGY

The intrinsic sensitivity of ion mobility spectrometry combined with distinguished enrichment properties of thermal desorption by ITEX DHS leads to outstanding possibilities in the field of trace analysis where very low detection limits are required.

INTRODUCTION

The In-tube Extraction (ITEX) Dynamic Headspace (DHS) Tool performs the enrichment of volatile or semi-volatile compounds during headspace analysis to increase the detection limits and concentrate compounds which cannot or barely be proven by common headspace sampling. By using a dynamic extraction method the compounds out of solid, liquid or gaseous phases can be easily enriched in the microtrap of the ITEX tool through continuously collection of the headspace. The ITEX trap is filled with an adsorbent material which can be application specifically adjusted and easily exchanged depending on the physical and chemical properties of the sample. Common trap materials are Tenax or activated charcoal. The reliability of the system is ensured by a closed design, where no sample loops, transfer lines or switching valves are used so that there is no risk of cross contamination, sample loss or carryover. The ITEX tool can be easily exchanged and the cycle times are optimized by overlapped sample incubation, active purging, trapping and cooling systems. The ITEX tool is already being used in several industrial fields for example for the analysis of food and beverages, flavors, drinking water, petrochemicals, polymers, pharmaceuticals and residual solvents.

ITEX DHS

Working Principle of ITEX DHS

1. The sample is heated and / or agitated in a sealed vial.

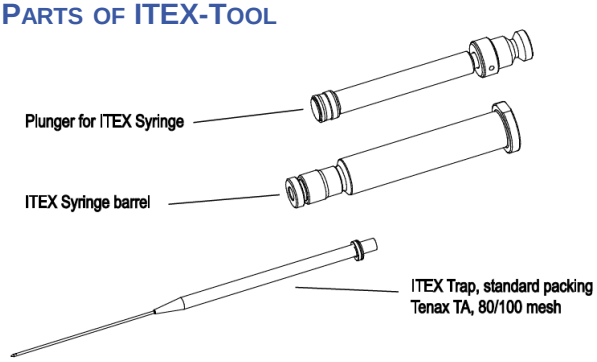
2. The ITEX needle pierces the sealed vial and the syringe pumps the headspace gas through the cold trap. Analytes are focussed on the sorbent material. This procedure can be repeated as many times as necessary in order to gain sensitivity. An additional step to remove water or other solvents from the trap can be added.

3. The loaded ITEX trap is flash heated up to a max. temperature of 350°C and analytes are desorbed into the hot GC injector.

4. After thermal desorption the hot ITEX trap is cleaned with inert flush gas.

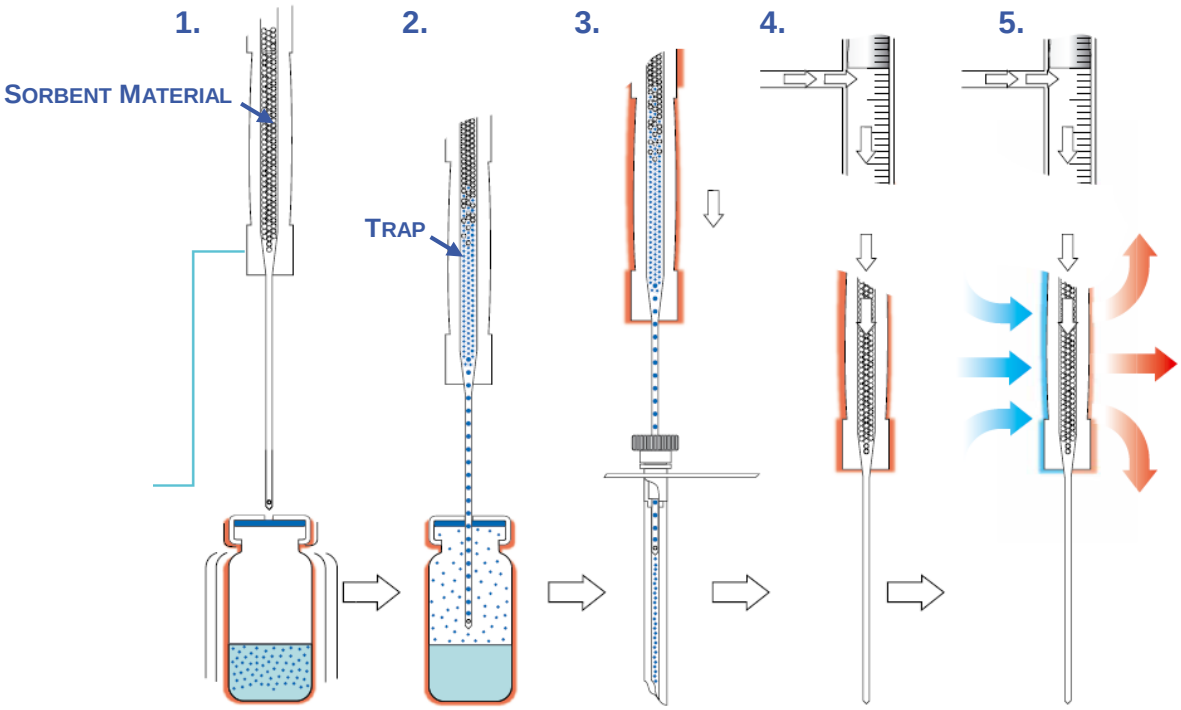
5. Active cooling and low thermal mass allows for short cycle times.

Parts of ITEX-Tool



Assembled ITEX-Tool & Park Station

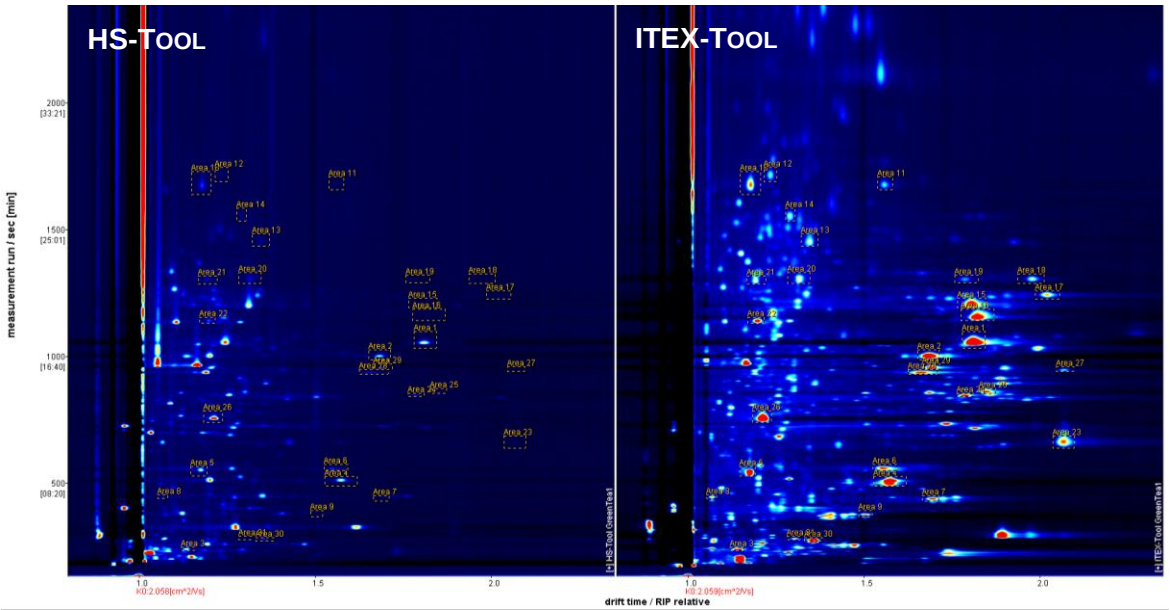
Working Principle



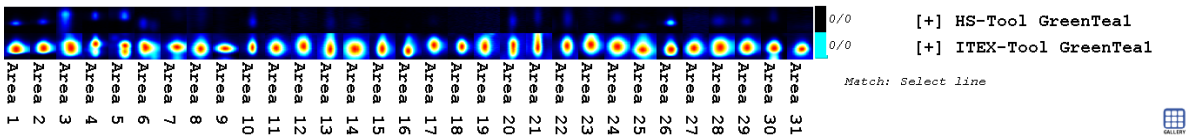
GREEN TEA

In the following green tea samples were analysed by HS- and ITEX-Tool in order to optimize a method for sensitive detection and analysis of green tea's fingerprint. Several signals, based on compounds ranging from the high to semi-volatile region´, were exemplarily selected and evaluated in order to depict the enrichment effect using additional ITEX option (marked by yellow rectangles)

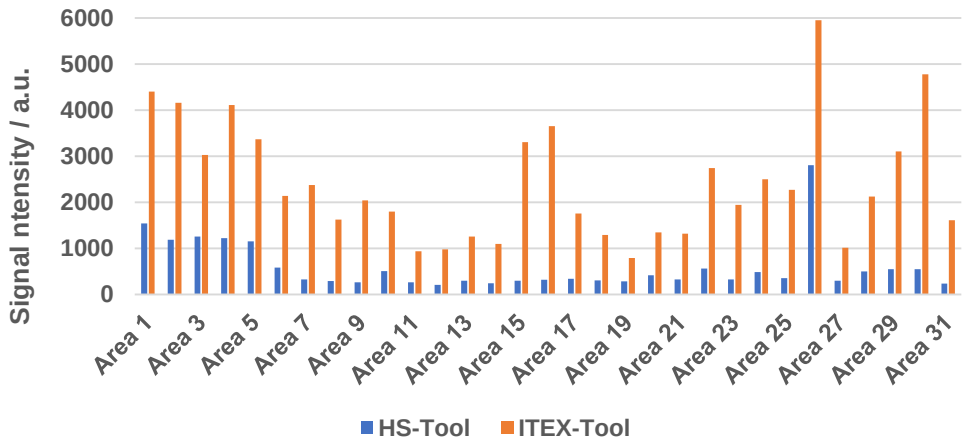
GC-IMS DATA OF SELECTED AREA SET



GALLERY PLOT OF SELECTED AREA SET



SIGNAL INTENSITIES OF EVALUATED SIGNALS



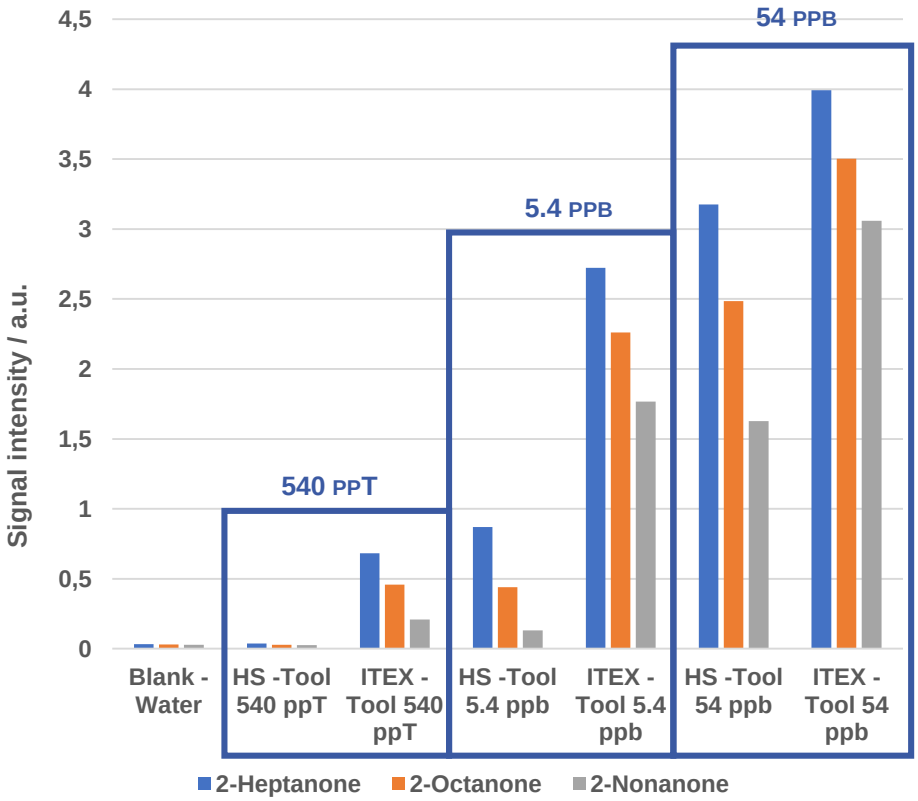
0.5 grams of green tea samples were prepared for headspace analysis

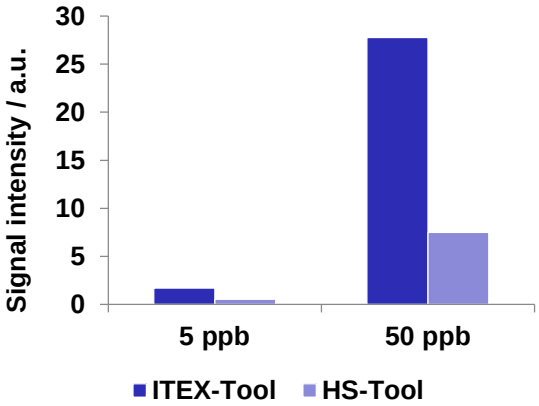
Flavour Compounds

Homologues Series of Ketones

In order to compare HS- and ITEX-Tool a dilution series of a homologous series of 2-Ketones was prepared and measured using both tools. The concentration of analytes in the solution was 0.54, 5.4 and 54 ppb.

Signal Intensities of a homologous Series of Ketones



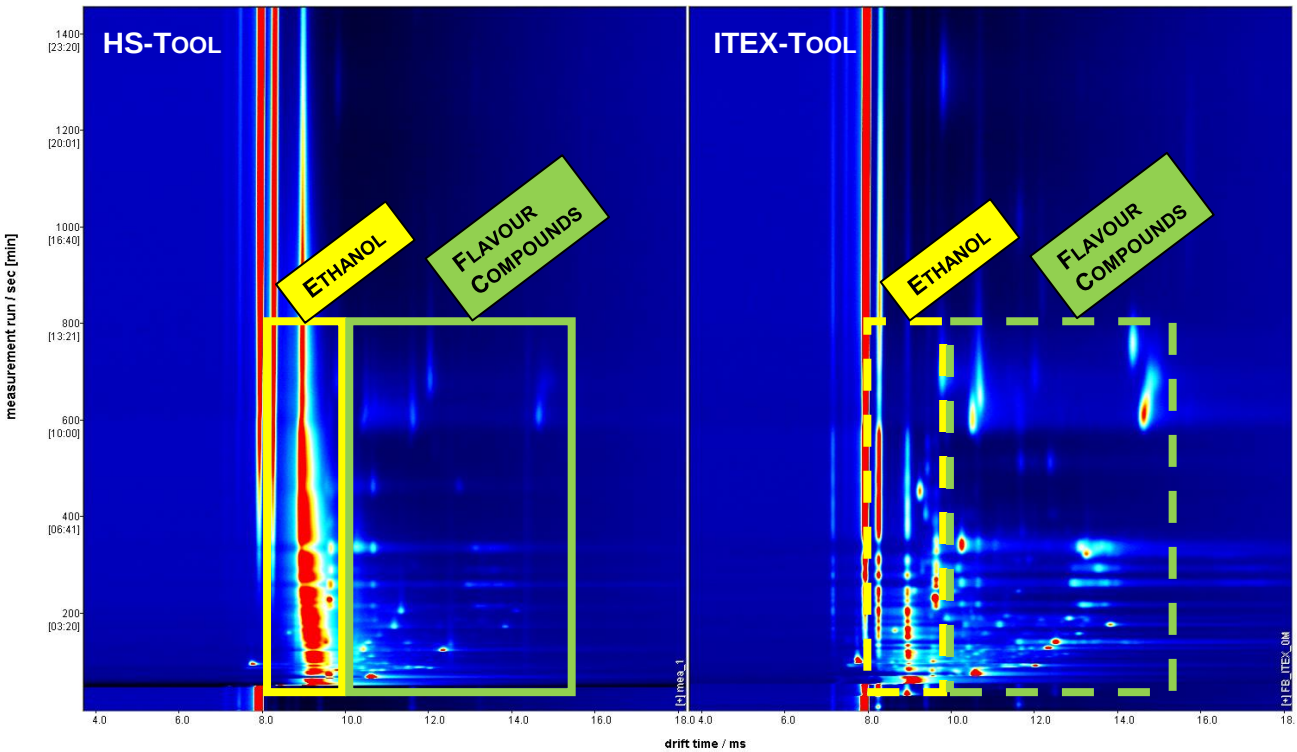


Guaiacol

In order to compare HS- and ITEX-Tool a dilution series of guaiacol solutions (aqueous, ddH_2O) were prepared at 5 and 50 ppb. Guaiacol is a natural occurring flavour compound exhibiting a characteristic smell and taste. It especially occurs in cured groceries, but is also one of the flavour compounds in whisky. The odour detection threshold lies in the single-digit ppb range.

ETHANOL REMOVAL / REDUCING PERTURBING MATRIX EFFECTS

The extraordinary high sensitivity of GC-IMS technology can be boon and bane at the same time. When analysis of traces is desired it definitely is a bane working with an IMS, but when it comes to samples exhibiting a high background matrix it also can be a boon. Usually gas chromatographic separation can solve these problems. In the following case increasing the gas chromatographic separation does not yield to acceptable results. GC-IMS data depict the headspace analysis of Fernet Branca, which exhibits a content of ethanol around 40 %. The high content of ethanol (signal marked by yellow rectangle, solid line) resulted in very high peak intensities and also undesired tailing of the signal, which perturbed detection of other compounds in the region of elution. As can be easily seen coeluting compounds were disguised by the ethanol peak.

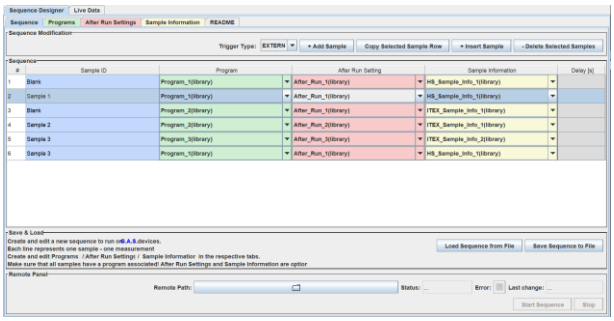


In order to get rid of the high ethanol content, which was introduced into the measuring system an extra *ethanol removal* step was introduced into the method supported by the ITEX-Tool. Between step 2 and 3 (see working principle) a preparation step, where temperature of the ITEX syringe was slightly increased and syringe was purged simultaneously. After optimization of the parameters the ethanol content could be drastically decreased (compare yellow solid and dashed rectangle), whereat masking of coeluting compounds also was dramatically decreased, so several signals appeared in the region of coelution (compare green solid and dashed rectangle).

LAB AUTOMATION – CHOICE OF PAL

CHOICE OF AUTOSAMPLER: PAL RSI OR PAL RTC

The standard G.A.S. FlavourSpec® comes with a PAL RSI autosampler and is easily upgradable by replacing the existing HS-Tool with an ITEX-Tool by hand. Using the *change tool* function of the autosampler this can be done within minutes. If an automated exchange is necessary the PAL RTC (Robotic Tool Change) is the right choice. The different PAL tools are placed in the included *park station* (up to three injection tools) if not in use. The robotic tool change function enables unattended 24/7 sample preparation and measurement. In combination with the *G.A.S. Sequence Designer* software the whole measurement campaign (incl. measurement sequence, sample information, measurement program and additional sample information) is prearranged on the PC.



G.A.S. *SEQUENCE DESIGNER*

Once started the whole sample preparation and measurement process is fully automated – incl. the exchange of tools (Headspace, ITEX, etc).

FLAVOURSPEC® - PAL RSI COMBINATION

FLAVOURSPEC® - PAL RTC COMBINATION

FLAVOURSPEC® - ITEX DHS COMBINATION

GC-IMS ITEX DHS TECHNOLOGY

The GC-IMS combines the high selectivity of a gas chromatographic (GC) separation with the extraordinary sensitivity (low ppb_v or µg/L range) of an ion mobility spectrometer (IMS). Therefore it is an excellent analytical tool, even for measurements in complex matrices.

Combining the intrinsic sensitivity of ion mobility spectrometry with distinguished enrichment properties of thermal desorption by ITEX DHS leads to outstanding possibilities in the field of trace analysis where very low detection limits are required.

OVERVIEW

- Combination of ion mobility spectrometry and ITEX technology yields to extremely sensitive detection limits
- Enrichment of volatile organic compounds (sensitivity low ppb- to low ppT-range)
- Several standard adsorbent materials commercially available
- Selective enrichment of analytes possible
- Also selective depletion of undesired compounds possible (water, ethanol, matrix compounds in high concentration)
- Fully automated sample handling

APPLICATIONS

- ✓ General enrichment of volatile organic compounds in order to gain sensitivity
- ✓ Selective enrichment/controlled depletion of compounds (in order to dispose undesired or perturbing matrix compounds)
- ✓ Highly sensitive detection of sample fingerprints
- ✓ Alcoholic beverages: Whisky, Wine, Liquors (aging, quality control, flavour analysis, e.g.)
- ✓ Detection of TMA in fish or shrimps
- ✓ Sensitive detection of Geosmin
- ✓ Detection of different ketones in food stuff

SPECIFICATIONS

Measuring technique	2-dimensional separation by GC-IMS technology
Enrichment technique	ITEX DHS
Detection limit	Typically low ppb- to ppT-range
Automation	PAL RTC exhibiting automated tool changes
Sample capacity	1 Rack: 60 vials or 2 racks: 120 vials (vial volume = 10 or 20 mL)
Sorbent material of ITEX tube	TENAX TA/GR, Carbopack C, Carboxen 1000, Carbosieve S III, Molecular sieve 5A, combinations/more materials on request
Injection tools	Headspace and/or ITEX
Incubator capacity	Up to 6 vials

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