Gas Chromatograph coupled to
Ion Mobility Spectrometer (GC-IMS)

When **Quality Control** becomes **impartial & easy** ...

- headspace analysis (no sample pre-treatment)
- sensitive (low ppb)
- NIST based library

INTRODUCING G.A.S. DORTMUND

Company

G.A.S. is a Dortmund based high-tech company founded in 1997 that has focussed on gas analytical solutions for specific customer question related to the measurement of Volatile Organic Compounds (VOCs). The interdisciplinary team of chemist, electrical, mechanical and software engineers stands out due to competence and experience related to the applied technology of Ion Mobility Spectrometry (IMS). Compared to other IMS manufacturers G.A.S. applies IMS in civilian application and adapts its instrument platform according to the specific analytical question of its customers.

- G.A.S. supports its customers concerning method developments for their analytical questions and has carried out hundreds of feasibility studies to prove the advantages of its technological set-up.
- G.A.S. provides analytical services in its facilities same as at customers' side.
- G.A.S. provides an outstanding support and after-sales service to its customers using its global distributor network.

Figure 1: G.A.S. facility at TechnologyCenterDortmund, Germany

Mission:
G.A.S. is a trend-setter in innovative analytical instrumentation that is reliable and extremely easy in use. Coupling its unique set-up of Ion Mobility Spectrometers to Gas Chromatographs G.A.S. provides significant value to its customers related to quality control related to volatile organic compounds.

Technology

G.A.S. has fundamental know-how in the field of IMS as it develops and manufactures its sensors, electronics, software same as pneumatics in-house and on a stand-alone base. Further to that a high-end hard- and software platform allows to adapt a general set-up according to the specific analytical measurement requirements and/or assignments, especially when it it comes to trace detection of VOCs..

IMS provide a qualitative information due to a specific flight time of each substance's ion and a quantitative according to the peak height/volume or area.

With a unique set-up of its heatable IMS the sensor reaches a resolution of R~100 (t/2Wh - drift time/temporal width at half length in ms) which avoids overlapping and allows an excellent peak identification and further substance quantification.

When it comes to rather complex matrices like found in food & flavour, process and environmental same as medical applications the coupling to a gas chromatographic (GC) pre-separation represents G.A.S. solution of first choice. By doing so an enhanced **2-dimensional separation** of GC plus IMS is achieved to still assure a reliable substance identification.
On the other hand the cutting-edge sensitivity of the IMS with detection limits of low-/sub ppb$_v$-level makes pre-concentration processes redundant and represents a significant difference to common detectors.

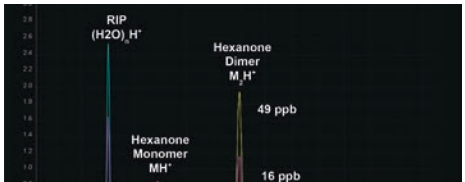


Figure 2: Specific IMS drift time provides qualtative (peak position) and quantitative (peak height, volume or area) information

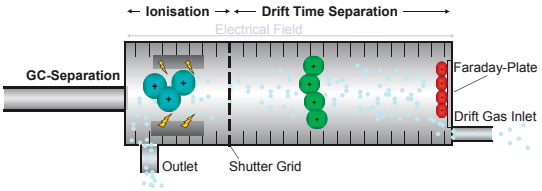


Figure 3: 2-D separation by GC and IMS allows to measure in complex matrices.

The 3-dimensional dataset ensures a compound identification using the specific gas-chromatographic retention time as well as IMS drift time while the measured intensity correlates to the concentration of the respective compound.

The data analysis is typically based on 'evaluation areas'. Analog to a range in a 2D chromatogram the area represents a specific compound position. Automatic determination of the compound signals intensities (signal height, peak-area and -volume) can be used for compound calibration/quantification and statistical evaluations.

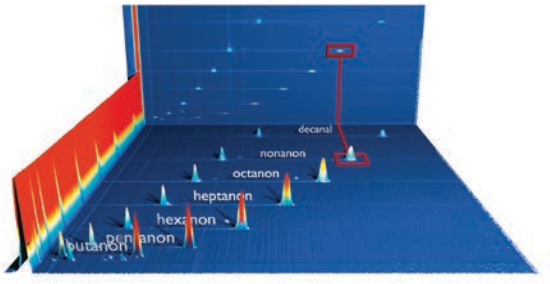


Figure 4: 3-D data allow a precise compound inditification and quantification.

Software

The **Laboratoy Analytical Viewer (LAV)** of G.A.S. offers a very attractive software package to make best use of the information provided by the chromatogram data that is compatible with most Windows based programs for data export if needed.
The LAV software suite and its application related 'Plug-ins' allow to follow two appoaches: The first comprises of the classical analytical methods to detect single compounds and/or their concentrations that are defined or known as markers for specific product characteristics. To work this way the user has to set-up calibrations in order to make decissions concerning unknown samples under testing.

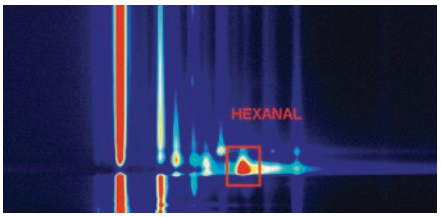


Figure 5: Determination and marking the compound of interest.

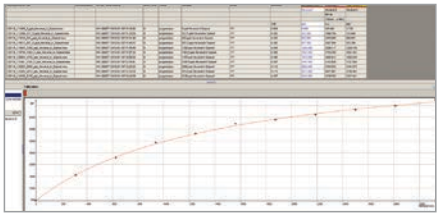


Figure 6: Calibration example for hexanal

The second approach follows the concept to look at the entire chromatogram respectively the peak pattern or 'fingerprints' as a result of the whole VOC composition of the headspace. Here the LAV offers smart Plug-in tools like the 'Reporter' to easily compare a reference sample with one or several unknown samples at a glance. Alternatively the user can easily analyse the presence or difference in intensities of substance peaks by the use of the 'Gallery-Plot' to easily determine classes of products or their variations which for example is very helpful for product authentication.

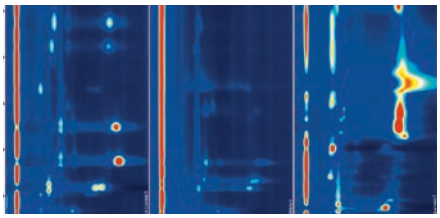


Figure 7: 'Reporter' Plug-in to receive results at a glance

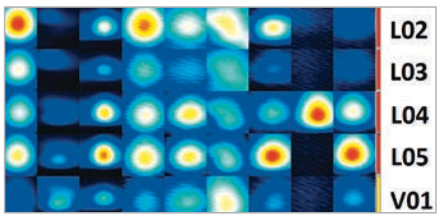


Figure 8: Gallery-Plot 'Plug-in' cuts out peaks that show differences (appearance, intensity)

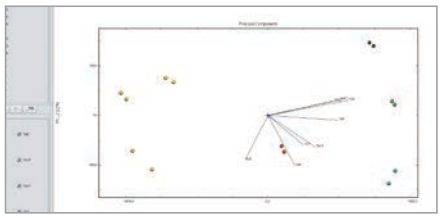
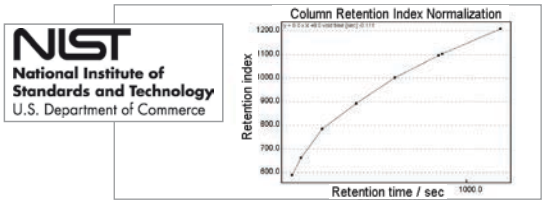


Figure 9: Principal Component Analyis for distinctive method development.

As a more advanced tool a principal component analysis, 'PCA Plug-in' is available to complete the software package for evaluating samples based on complex information where intensities are automatically extracted from measurement results. The dataset for the PCA are the intensities of evaluations areas for multiple samples.

Sophisticated software tools to further compare and classify samples easily:

For identification of unknown compounds normalization of the G.A.S. devices with respect to retention index is possible. Click-to-search for matching compounds in the NIST 2011 Retention Index Database is provided. Further to that compound identification based on IMS drift time index libraries is possible for a continuously growing number of compounds. Both retention- and the drift time index libraries can be enlarged by the customer.



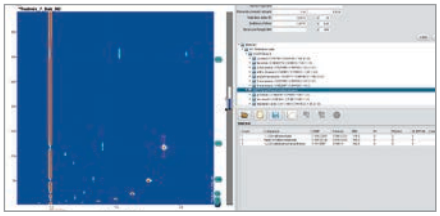


Figure 10: GC (retention index) x (drift time) libraries supported compound identification.

APPLICATION FIELDS

Food & Flavour

Product identification and authentication same as quality control in general plays a more and more important role every day especially in the food and flavour industry.

IMS in combination with a gas chromatographic pre-separation and when equipped with an automatic headspace sampler for an easy and reliable sampling stands out with unique characteristics in several quality control related applications. Due to the physical working principle of the GC-IMS technology systems are extremely reliable and set up with a result orientated and very easy-to-use menu. Their sensitivity (low ppb/µg/m³) for volatile organic compounds (VOCs) lies in the range of the human nose for many applications so that impartial results can be provided to e.g. support sensory panels and avoid time and labour-intensive analysis when compared to working with GC-MS

Figure 11: Food products can directly be analysed without pre-treatment

By headspace analysis of solids and liquids the FlavourSpec® either identifies and quantifies a single compound/marker or the whole peak pattern found within the 2-dimensional GC-IMS chromatogram to quickly and result orientated test for:

- Food freshness, storage conditions, best before date
- Quantification of diacetyl and pentanedione during the beer brewing process
- Quality control of commodities and finished products:
  - Ingredients and herbs
  - Olive oil [1]
  - Hops [2]
  - Fish, meat
  - Fruit juices and other mixtures
- Product authentication (habitat, detection of frauds, counterfeits)
- Impartial prove of product flavours
- Support of sensory panels
- Early Detection of off-smells
- Flavour composition, blending (green tea, coffee, cigarettes etc)
- Optimisation of manufacturing processes, intermediate products not made for human testing
- Outgasing VOCs from packaging/plastics/papers
- ...

Figure 12: Quality control of green tea: Authentication, habitat, quality grade and storage time

FlavourSpec®:

- Measures flavour inducing VOCs with a heteroatom
- Manual injection or automatic sampling (no pre-treatment needed)
- Does not require lab environment, can work at-line
- Extremely easy-to-use (colleague-to-colleague)
- Rugged: No maintenance required
- Direct sampling: No pre-treatment needed

Figure 13: FlavourSpec®

[1] Garrido-Delgado, R., Arce, L., Valcarcel, M. (2012). Multi-capillary column-ion mobility spectrometry: a potential screening system to differentiate virgin olive oils . Anal. Bioanal. Chem., 402, 489-98.

[2] Kurzweil P., Hildebrand A., Riedmayr A., (2013), Hopfensorten und ihr Aroma, Nachrichten aus der Chemie, Juni 2013, 656-661

Breath Analysis

Breath analysis represents a diagnostic technique which can provide information beyond conventional analysis of blood and urine e.g. NO-testing for asthma. Advantages of breath tests are that they are non-invasive, painless and do typically not require trained personnel for sampling.

Hundreds of different substances are present in human breath depending on nutrition, metabolic state including diseases and medication, microbial infections and personal oral hygiene. The appearance or absence of specific metabolites can be used for early diagnosis and their presence for therapy control when identified relevant for a particular disease or medication.
Several research studies show that gas chromatographs-ion mobility spectrometers (GC-IMS) due to their selectivity and sensitivity are suitable instruments for measuring volatile organic compounds (VOC) in exhaled air [1] that are related to intoxications and diseases [2].

Figure 14: BreathSpec® during operation

Application related to the human breath are:

- Recognition or monitoring of diseases through VOC testing
- Monitoring of work-related exposure of hazardous substances, personal safety
- Control of drug decomposition (Pharmacokinetics)
- Outgasing from human skin

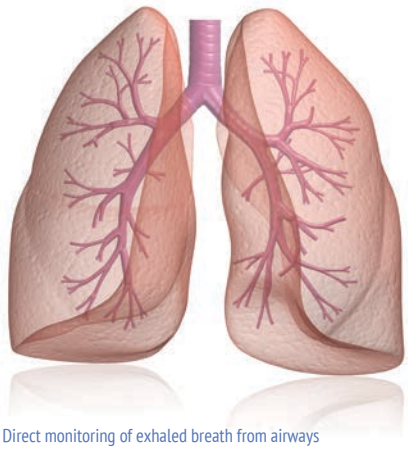


Figure 15: Direct monitoring of exhaled breath from airways

BreathSpec®

- Integrated medical spirometer with disposable mouthpieces for easy and reproducible sampling (flow, CO2/O2 control)
- Heated sampling lines to clean instrument and avoid memory effects
- Versatile due to 3 sampling modes (continuous exhaling, collection of sample during interrupted exhaling, suck-in of sample through calibration port)
- Integrated pump and 6-port-valve for manual and automatic sampling of gases
- Portable
- Automatic data acquisition
- Extremely easy operation by touch panel

Figure 16: BreathSpec®

[1] Ruzsanyi, V.; Baumbach, J.I.; Sielemann, S.; Litterst, P.; Westhoff, M.; Freitag, L.: Detection of human metabolites using multi-capillary columns coupled to ion mobility spectrometers, J. Chromatographia A 1084 (1-2) (2005) 145-151

[2] Nikolaos Pagonas equal contributor, Wolfgang Vautz equal contributor, Luzia Seifert, Rafael Slodzinski, Joachim Jankowski, Walter Zidek, Timm H. Westhoff, Volatile Organic Compounds in Uremia, Plos one, September 25, 2012

APPLICATION FIELDS

Environment & Process Industry

Monitoring of ambient air with respect to industrial gases, contaminants or VOCs causing bad smell become more important to the industry due to a more demanding environmental legislation and cost-by-cause principle. Therefore e.g. stack monitoring or odour mapping are assignments where sensitive, reliable and rugged analytical instruments that go beyond common sensor technologies are in need. Besides that instrument set-up requires a high separation capacity to avoid interferences e.g. with moisture and outstanding specificity at the same time if the VOC source should be identified.

G.A.S.' systems can be adopted to various environmental and process control related applications by adjusting sampling, operating mode as well as data analysis in a way its customers can take maximum benefit from. Robustness, rare need of maintenance (check ups) and the easiness of operation are the premises for industrial solutions. Smart and straight forward menu, learnable in few hours without the need of an analytical background complete G.A.S. on-site focussed approach. By that it becomes feasible to transfer the analytical lab to the place where instant and reliable results are needed.

Control of siloxanes in biogas: The monitoring of processes like biogas quality as fuel to produce heat and electricity is essential because due to the presence of washing agents, cosmetics, skin care products, silicone oils etc the presence of different siloxanes is induced. When the gas contains a certain amount of siloxanes and is combusted to generate power siloxanes are converted to silicium dioxide (SiO_2). Corrosion and system break downs are possible consequences.

Figure 17: On-site detection siloxanes in biogas

Monitoring of **Odorants in natural gas** like the sulphur-free Gasodor®-S-Free® or the sulphur containing compound tetrahydrothiophene (THT) have to be assured and controlled by the power utilities to secure the alarming smell of leaking natural gas. The GC-IMS operates with a tailor made menu, automatic sampling and data output so that a concentration control of the odour admixture can be secured by a technician without special analytical expertise.

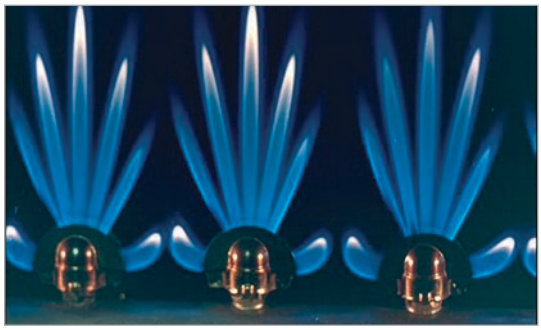


Figure 18: Testing for odorants in natural gas

Industrial products can under certain circumstances be contaminated with traces of **impurities**. In case of low olfactory thresholds these impurities can respresent off-smells that affect the quality of the product from a customer's point of view and therefore need to be monitored.

Environmental applications are:

- Process control (siloxanes in biogas, filter efficiency)
- Quantification of odorants in natural gas (THT, Gasodor-S-Free)
- Finished product control (impurities in solvents, unwanted smell from production)
- Stack on-line monitoring
- Quality control of (intermediate) products not made for human consumption/testing (chemical industry)
- Clean room monitoring (NH3, HCL)
- Maximum allowable concentration of toxic industrial chemicals (e.g. dimethylsulphate)

GC-IMS:

- Integrated pump and 6-port-valve for manual and automatic sampling of gaseous samples
- Portable
- Rugged: Suitable for on-site applications
- Results can be displayed on touch-screen or transferred via analogue output, Ethernet, USB

Figure 19: GC-IMS

Ion Mobility Spectrometer as OEM-Module

Hybrid solutions offer new dimensions in analytical fields

The stand alone plug-and-play IMS detector as OEM module by G.A.S. mbH enables versatile use of the technology according to individual application requirements. Besides the advantage of reasonable costs compared to fully equipped IMS instruments, this modular set-up allows configure the analytical system around according to customer's needs. The device can be coupled to standard GC systems or the user can alternatively use a membrane inlet system or even thermo desorption unit like SPME or needle trap.

Figure 20: Benchtop GC with IMS as sensitive detector

Samples are ionized by using a tritium source which intensity lies below the exemption limits of the EU directive 29/96 EURATOM. Power input is 24 V DC and the data output is realized by an USB 2.0 high speed digital interface. For the introduction of drift- and carrier gas (usually nitrogen or synthetic air) 3 mm Swagelok connectors are used. The sample is introduced using a 1/16" sample line. The modul can be heated up to 100 °C and has a resolution of ~ 100.
The parameters of the modul are controlled by an external PC-software belonging to the scope of supply.

Advantages of the IMS detector are:

- Sensitive for VOCs with heteroatoms like ketones, aldehyds, alcohols, amins or halogenated and sulphurous compounds etc.
- Detection limits in the low ppbv (µg/m³) range
- Selective due to characteristic analyte ion drift times
- No licence for source required according to EU directive 29/96 EURATOM
- High reproducibility < 3 % for peak intensity and < 1 % for drift times
- Operation gas nitrogen or synthetic air
- Works at ambient pressure
- Compact design
- Easy to use (plug-and-play)
- Free of maintenance
- Stand alone software for data aquisition and software suite for 3D GC-IMS data analysis
- Positive and negative ions drift voltage (switchable)

Further specifications of OEM-module:

- DC input: 24V
- Signal output: analogue or digital
- Gas connections: 3 mm or 1/8" Swagelock

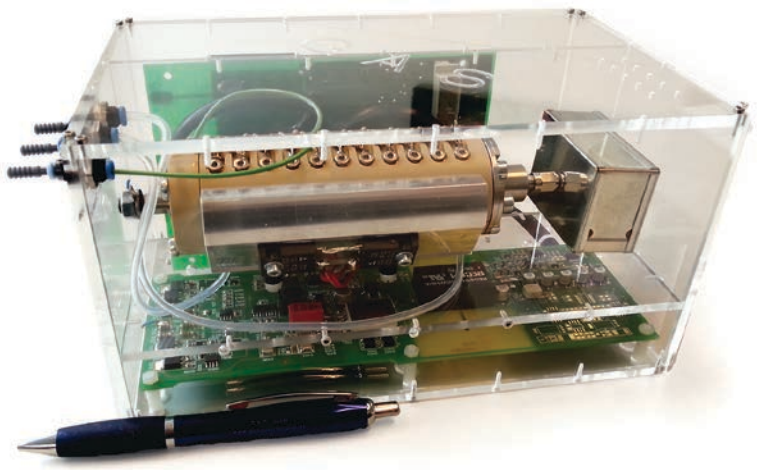


Figure 21: OEM-Module of IMS

PRODUCTS

G.A.S. cutting edge instruments stand out due to several advantages concerning analytical applications, like:

- Ionisation source below exemption - no licence required according to EU directive 96/29/EURATOM.
- Generate positive and negative ions by one-click
- Outstanding sensitivity (ppb-/µg/m³)
- Do not need any sample pre-treatment
- No special skills required and very easy to operate
- Storage of acquired data on the device and/or data transfer to PC
- Identification of individual compounds through library (via NIST GC-retention indices and IMS drift times) or fast decision making via automated classification through 2-dimensional peak pattern 'fingerprint' analysis
- Fast run times (inject-to-inject <15 minutes)
- N_2 or synthetic air as consumable for operation
- No wear parts
- Low maintenance (every 24 month routine check-up)
- Cleaning mode in case of contamination: >100°C
- High Reproducibility through physical working principle: >3%

Table 1: Overview of IMS systems by G.A.S.

	A-IMS	UV-IMS	GC-IMS	FlavourSpec®	BreathSpec®	OEM-Module
Source	3H	UV-10.6 eV	3H	3H	3H	3H
Heated Gasways	Isotherm up to 80°C	No	Isotherm up to 80°C	Isotherm up to 80°C	Isotherm up to 80°C	Isotherm up to 80°C
Sampling	6-Port-Valve Direct	6-Port-Valve Direct	6-Port-Valve	Heated Injector 250°C	Medical Spirometer Through Calibration Port	Direct
Gas Supply N_2 or N_2O_2	External Internal circuit	External	External Internal circuit	External	External	External
GC-Column	No	No	Multi-capillary-column Standard FS-column (up to 60 m, isothermal heated)	Multi-capillary-column Standard FS-column (up to 60 m, isothermal heated)	Multi-capillary-column	Designated for versatile coupling
Measures	Gaseous VOCs	Aromatic Compounds	Gaseous VOCs in complex matrices	VOCs in headspace of liquids and solids	VOCs in human breath (intoxication, markers for diseases)	Gaseous VOCs
Matrices	Defined, homogeneous matrices	Defined, homogeneous matrices	Complex gaseous samples	Complex headspace composition	Complex human Breath	N. a.

Customers who trust us:

- Akzo Nobel Chemicals bv, NL
- Applied Mass Spectrometry Laboratory S.L., ES
- Beth-El Zikhron Ya'aqov Industries Ltd., IL
- Clariant Produkte Deutschland GmbH, DE
- Daimler AG, DE
- Department of intensive Care of the University of Innsbruck, AUT
- Dortmunder Energie und Wasserversorgung GmbH, DE
- E.ON Bayern AG, DE
- E.ON Hanse AG, DE
- EADS Deutschland GmbH, DE
- Federal Institute on Environmental Affairs, DE
- Federal Office of Industrial Safety and Occupational Health, DE
- Firmenich SA, CH
- Food Industry Research and Development Institute, TW
- Hydro Aluminium Rolled Products GmbH, DE
- Intersnack Knabber Gebäck GmbH&Co. KG, DE
- Leibniz-Institut für Analytische Wissenschaften - ISAS - e.V.
- JT International Germany GmbH, DE
- Marmara Research, TR
- Ministerio de Ciencia e Inovación (CSIC), ES
- NeoMediTec, P
- Nestlé SA, CH
- Nordia, JAP
- Odournet GmbH, DE
- Radeberger Gruppe KG, DE
- Riken Keiki Co., Ltd., JAP
- RWE Service GmbH, DE
- SENSORTEC Ltd., NZ
- SGS Nederland B.V., NL
- Stadtwerke Trier GmbH, DE
- SYMRISE AG, DE
- Technical University Berlin, DE
- Tianjin Entry-Exit Inspection and Quarantine Bureau, CN
- University of Cordoba, ES
- University of Iasi, ROM
- University of Kuopio, FIN
- University of Lodz, PL
- University of Northumbria, GB
- Zhejiang University, CN

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