



MPS PrepStation in the LAVES laboratories. Due to its "wing span", the staff refers to it as "Condor". The MPS guarantees uniform results when generating standard solutions even when operated by different users.



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LAVES: The first and foremost address in the state of Lower Saxony, Germany, for monitoring food safety including pesticide levels in food.



Laboratory on-site: Visiting LAVES in Oldenburg, Germany

On the Wings of the Condor

The State Office for Consumer Protection and Food Safety (LAVES) in Lower Saxony, Germany, came out on top with the best results in a Europe-wide round robin test of pesticide laboratories for the determination of pesticides residues in cereals. In the words of the President of LAVES, Prof. Eberhard Haunhorst, Ph.D., this achievement is made possible by highly educated, trained, and experienced staff. While obviously true, there is more to the story: In addition to know-how and experience, top notch laboratory performance requires the right laboratory equipment. GERSTEL Solutions Worldwide magazine visited the LAVES pesticide laboratories where we joined the staff at work to gain some insight.

By Guido Deussing

The room has an air of food storage just like you would experience in a large scale kitchen facility. A whiff of ocean is mixed with a tangy earthy smell. Yesterday it was fish, today mushrooms are on the menu. These are delivered straight from the farm to the table: Mushrooms, oys-

ter mushrooms, wood ear / Judas ear, and shiitake in little black and red baskets are lined up across the large table. However, the proof of these foods will not be in their eating – they are here to have their pesticide residue levels determined by a leading European pesticide laboratory.

Among the best laboratories for pesticide analysis in the EU

The Lower Saxony State Office for Consumer Protection and Food Safety (LAVES) has its headquarters in Oldenburg (OL), Germany. Here, the main laboratory is located, part of a network of six laboratories throughout the state. The mission statement makes it clear: “LAVES – we act for the collective good of both humans and animals”. Among the bullets in the Mission Statement, may be a hint as to why the laboratories are so successful: “LAVES is committed to continuously improving its processes and achievements”. Among its guiding principles is to offer full transparency and to be beyond reproach, always delivering the right results. Harmful food must never be allowed to reach the plate of the consumer. The attitude is sensible, but can it always be ensured? Reality may not always be as clear-cut even though the law is clear: Consumer safety must always be put before business interests. LAVES uses the most modern analytical techniques and equipment to monitor and test food and feed for compliance, forming a key pillar in the consumer safety structure of Lower Saxony, a German State with nearly 8 million inhabitants. The LAVES headquarters are housed in a spacious multi-storied glass covered building and harbors a leading European



GC/TOF-MS for pesticide screening: Thanks to the GERSTEL MPS with Automated Liner Exchange (ALEX), even series of “dirty” samples are easy to analyze.

pesticide laboratory for food and feed analysis. In charge and responsible for its performance is Iris Suckrau, Ph.D., a sprightly food chemist who has learned her trade step by firm step starting with vocational training as a Laboratory Technician followed by a High School Diploma and a Food Chemistry university degree capped with a Ph.D. Talented, hardworking, and ambitious, Dr. Suckrau has radiated her infectious positive energy at LAVES since



Team members: TOF, Casper, Ernie, Bert – every GC/MS System in the LAVES pesticide lab has been given a nickname.

1995, hunting for dioxin in food and feed for fifteen years before joining the pesticide laboratory. Four Scientists and 15 Technicians work in the laboratories. “When I started here, there were only four of us”, she reminisces, relaxing at her neatly organized desk in an office that shows no sign of clutter. Dr. Suckrau spends as much time there as in the laboratory, going over analysis protocols, reports, and interpreting results as well as generating expert assessments. “What we generate must be legally flawless and incontrovertible in court”, the scientist says, “we do not work for companies or private persons, but solely for the government and public services, our clients are food safety monitoring agencies, counties and towns”. The 3,000 food and feed samples received annually by LAVES are generally submitted at the behest of the government.

Food analysis mainly of seasonal produce

In earlier years, says Iris Suckrau, food inspectors were asked to each deliver a specific number of food samples to the laboratory for testing. Some clever inspectors went straight to the nearest supermarket produce department and took samples of every tropical fruit and orange in sight, expecting them to be laden with pesticides and likely to earn the inspector praise. “They of course were able to submit their assigned number of samples in no time at all”, says Iris Suckrau, “but from a consumer safety and testing stand point it didn’t make much sense. Incidentally, tropical fruits are much better than their reputation”.

Times have changed, though, random testing is out. Nowadays, samples are increasingly taken on a risk-assessed basis with sweeping regional controls in our own area, says Iris Suckrau: “A key focus is on seasonal produce such as asparagus and strawberries; we are, so to speak, keeping our own house in order”.

Whenever Greenpeace publishes new figures that point to increasing levels of chemical residues in bell peppers, lettuce and similar produce, the workload goes up at LAVES. Sensitized by the news, food inspectors look a little closer



On the same wavelength: Iris Suckrau (left) and GERSTEL have an ongoing collaboration. The first conversation with Sales Manager for the German speaking territories Michael Gröger (right) was about the first Cooled Injection System (CIS) GC inlet, installed at LAVES in 1997.

during unannounced visits to see if hygiene regulations are adhered to and quality standards met. More samples are then taken when visiting bakeries, meat producers, food processors, food merchants, cafes, restaurants, and other large kitchen facilities. If the food quality seems lacking, and if even there is the slightest suspicion of a potential violation of consumer protection laws and food safety regulations, the inspector must take replicate samples. The initial sample is sent to LAVES and the replicate sample is sent to the producer enabling them to request a second opinion from an independent third party certified laboratory in case LAVES confirms that residue levels are indeed too high. Since such tests are costly, typically the case first goes to court and a lawyer gets involved. “The first thing the lawyer defending the company found in violation does is to perform a detailed analysis of whether the sampling was performed in strict accordance with all protocols”, says Dr. Suckrau, “and because sampling procedures are both intricate and highly regulated, we train our inspectors on a yearly basis”. Many inspectors are trained bakers, cooks, or other food processing professionals that have gone through extra training as food inspectors. Unless the sampling process is performed correctly Dr. Suckrau and her team can never successfully do their job.

A look behind the scenes at the Pesticide laboratory

Hissing and clattering fills the room we enter, shielded from direct sunlight by louvers outside the windows. The interior of the GC/MS lab offers a certain familiarity to anyone who feels at home in gas chromatography. Neat rows of GC/MS systems are lined up on clean lab benches. Behind them, cables and gas lines run to the ceiling to their respective connection points. Exhaust ducts hover above the instruments. But the important thing for laboratory performance is how the instruments are equipped: Most of the GC/MS systems have a GERSTEL MPS mounted on top, outfitted with different options and ac-

cessories for comprehensive automated sample preparation and introduction. The introduction techniques range from liquid injection and Large Volume (liquid) Injection (LVI) to Headspace and solid phase micro-extraction (SPME). The QuEChERS extraction method (Quick, Easy, Cheap Effective, Rugged and Safe), is widely used for pesticide analysis, and is part of the daily routine in the LAVES pesticide laboratories. The analyzed QuEChERS extracts often contain a significant amount of matrix residue. Excess matrix residue deposited in the GC inlet liner can lead to changes in analyte recovery and inaccurate results. In order to ensure system stability, QuEChERS extracts are injected using the GERSTEL ALEX option (Automated Liner Exchange), replacing the GC inlet liner at user defined intervals, in this case after every 20 injections. Matrix residue is thereby automatically removed from the analysis system, enabling unattended analysis of large batches of samples in uninterrupted sequences overnight as well as on weekends and holidays. Thanks to ALEX, strawberry extracts and other complex matrices don't pose a problem to system stability.

Dr. Suckrau walks over to the GC Time-of-Flight Mass Spectrometer (GC/TOF-MS), which is used to run the first tests on a sample: “Screening with GC/TOF-MS and LC/TOF-MS delivers important clues as to the presence of analytes of interest to us in the sample”, she explains, “this information then enables us to find the best way forward in order to determine, for example, the pesticide residue levels as per the EU Regulation 396/2005.”

Analyte quantification is then performed using a four point calibration curve. If Maximum Residue Levels (MRLs) are exceeded, the analyst proceeds in well-defined, transparent and traceable steps using standard addition: The pesticides of interest are added to the individual samples in known concentrations. “Speaking of standard addition”, Dr. Suckrau suddenly says, “follow me!” The scientist marches towards the window area, takes a sharp right turn at the end of the bench, and stops in front of a GERSTEL



LAVES has a huge arsenal of standard substances at its disposal. These are used to generate dilution series, for quantification and reference purposes.

MPS XL PrepStation in Dual Rail configuration. “Due to its wingspan, we call this MPS stand-alone WorkStation the Condor”, explains Katja Kruse, a co-worker of Dr. Suckrau, “it holds a large number of standards and solutions in refrigerated trays for extended storage stability”. “The PrepStation“, Dr. Suckrau explains, “is extremely important to us“. European Union (EU) regulations for method validation and quality control in pesticide analysis require us to validate our results. To this end, LAVES stocks an arsenal of hundreds of reference standards, which can be used to generate standard solutions, mixtures, and associated dilution series. “As has clearly been established”, the scientist continues, “the standard addition technique is the best possible method when it comes to confirming and validating analysis results exceeding the specified MRLs. And this applies to both GC/MS and LC/MS”. Before “Condor” was given its well-deserved place in the lab, a lot of time was spent on preparing standard solutions, but the quality of these standards was not always sufficient to obtain final results of the highest quality. “Using the MPS PrepStation, we not only work more efficiently, we reliably

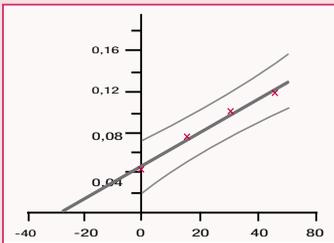


For improved quality and productivity leading laboratories like LAVES produce validated standards and automate their sample preparation using the GERSTEL MultiPurpose Sampler (MPS). Pictured Katja Kruse (left) and Iris Suckrau.

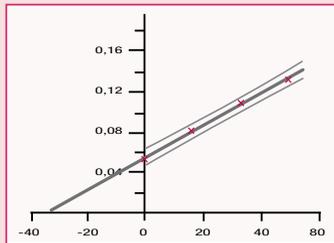
generate accurate and highly reproducible results”, says Iris Suckrau, “and that is extremely important, especially for a public agency that needs to deliver irrefutable evidence for court cases to help enforce consumer safety standards”.

Laboratory robots are only human...

Incidentally, all GC/MS systems in the room have been given nicknames: They are called Casper, Ernie and Bert, and it seems not only out of deference to “Sesame Street”: “By giving individual systems clearly recognizable names, it becomes easier to keep an eye on both the instrument and the task assigned to it”, according to Dr. Suckrau. Maybe we humans have a propensity for assigning personal traits and identities to robots and machines, as assistants and friends.



Manual Standard Addition



Standard Addition using the MPS

Automating the standard addition process helps on two fronts: Efficiency and performance. Shown to the left: The reproducibility resulting from manually generated standard addition, using pipette and dispenser. Shown to the right: Improved reproducibility resulting from automated standard addition using the GERSTEL MPS. Source: LAVES, Oldenburg, Germany.

Accurate Transfer of Liquids for highest precision using a bench-top Workstation and Accurate Add

GERSTEL
AppNote 187



AppNote 187

Automating the Accurate Transfer of Highly Volatile to Highly Viscous Liquids using a Bench-top Workstation www.gerstel.com/pdf/AppNote-187.pdf

Highly accurate standard addition and generation of standards and dilution series can significantly improve the quality of analysis results. Several useful tools and techniques are presented and evaluated, including thermostating; weighing; Accurate Add function for improved sampler precision; and vial venting to eliminate vial pressure build-up.

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Example: ALEX-GC-MS/MS-System for QuEChERS, Metabolomics, and Liquid Sample Prep