

Always one step ahead

Chemical production and reaction processes, as well as storage and transportation of the resulting products, represent a risk potential. Leaks into the environment, or unwanted exposure of workers to contaminants, must immediately be rectified. The law provides minimum requirements on how to proceed in such a case. A key role is played by instrumental chemical analysis, which is used by Public Health authorities and industry alike for monitoring air, soil, and water for potential contamination. GERSTEL Solutions Worldwide magazine visited the Ecology Services Laboratories of DowDuPont at the Chemistry Park Stade in Germany. The lab is responsible for water and environmental analysis at the site and we had a look around.

By Guido Deussing

A little less than one hour by car west of Hamburg, Germany, on the Southern banks of the lower Elbe river, lies the old Hanseatic town Stade, population 46,000. Stade is a county seat in the state of Lower Saxony. Right next door is the “old country” area, which is known for its many orchards and abundant supply of fruit and also serves as recreational area for the people of the region – as well as a flood area when the Elbe rises much



Dow combines the power of science and technology to passionately innovate what is essential to human progress. The Company is driving innovations that extract value from material, polymer, chemical and biological science to help address many of the world's most challenging problems, such as the need for fresh food, safer and more sustainable transportation, clean water, energy efficiency, more durable infrastructure, and increasing agricultural productivity. Dow's integrated, market-driven portfolio delivers a broad range of technology-based products and solutions in high-growth sectors such as packaging, infrastructure, transportation, consumer care, electronics, and agriculture. The Dow Chemical Company („Dow“) is a subsidiary of DowDuPont (NYSE:DWDP). DowDuPont was formed by Dow and DuPont to create three strong, independent, publicly listed companies in the areas of Agriculture, Material Science and Specialty Products.

above normal levels. The Schwinge, an Elbe tributary, runs through Stade. The meadows along the upper Schwinge and those in Stade along the rivulet are protected nature reserves due to both their biodiversity and their serene beauty. Here you find plenty of agricultural land and picturesque villages with quaint names that reinforce the impression of visiting unspoiled countryside. The impression is accurate, but there is more to the story. Stade may be in the countryside, but it is an industrial hub, home to global players. Airbus has produced airplanes

Among other things, they produce the vertical stabilizers (fins) for all Airbus aircraft. Around Airbus, a reliable supply chain of industries has developed.

The Dow Chemical Company also established a presence here. Since 1972, the largest chemical producer by revenue in the world has been producing in Stade at the address Bützflether Sand, which to Germans sounds like the name of the local beach on the river. Over time, Dow transformed its huge premises to an industry park and opened it to outside companies. The conversion was part of a quest to achieve higher efficiency: Share resources, develop and use synergies, and reduce cost by spreading the burden of expenses on multiple shoulders. The concept seems to work. Multiple companies are acting in synergy and coexisting at Bützflether Sand: Trinseo is operating a Polycarbonate facility; Olin is producing epoxides and chlorinated organic compounds; Air Liquide produces and supplies process-relevant technical gases; Air Products supplies hydrogen; Evides handles water treatment; Talke Carriers handles the central stock facilities; and the nearby Elbe Port serves as their gate to the world. Well connected to the freight depot train station and the container terminal, an almost perfect scheduling can be achieved for just in time transport of important goods, raw products and final products. These are shipped in all directions by rail, which unlike the express freeways in the area are generally unclogged. The industrial infrastructure here has been developed and perfected over decades, including freight forwarders that are specialized in the handling of hazardous goods. These and many other aspects make the Chemical Park Stade stand out as extremely well connected, even in global comparisons, though it is embedded in the countryside. The Ecology Lab in Stade is also in a good place: This environmental laboratory receives a lot of attention globally within the Dow organization because it, among other things, has developed outstanding methodology for monitoring volatile organic compounds (VOCs) using Thermal Desorption (TD).

here since 1959. The 2,500 Airbus employees in Stade are specialized in the production and development of carbon fiber reinforced materials for aircraft and space craft.



The Ecology Lab at Dow in Stade rely on the Thermal Desorption System (TDS) from GERSTEL for their TD analysis work. And for many other applications that are performed in the lab, GERSTEL instruments and systems play a key role. Reason enough for me to visit and to look behind the scenes. I made the appointment with Michael Gröger, GERSTEL Sales Manager for Germany, Austria and Switzerland and account manager for Dow. Michael has known and looked after the Dow colleagues since... forever and he is here for a meeting with my hosts Sandra Hirsch and Andreas Köhler, who are established experts in matters environmental and water analysis at the Ecology Lab. They are considering investing in a GERSTEL MultiPurpose Sampler (MPS) WorkStation to be used for automated generation of analysis standards. At this point, some questions remain open and my scheduled visit to the Ecology Lab offers Michael Gröger a good chance to join and provide some answers. We are meeting shortly after ten on a Wednesday morning in January in light snowfall. Sandra Hirsch will pick us up in her car at the visitor parking area. We cannot access the Chemistry Part Stade premises in our own vehicle. As outside visitors, a pre-arranged permit is needed even though Michael Gröger has paid regular visits for more than 16 years. Everything follows set rules. We need the permit and that requires time and patience. First time visitors – and those who haven't been back within the past year – must acquaint themselves with the safety rules by watching a video while the person at the reception fills in the paperwork and secures personal information from the visitor's ID card. After being registered in the Dow system, it is time for the test: Three questions about security procedures in the Chemistry Park must be answered correctly. The computer delivers the result on a small piece of paper that I hand to the receptionist who checks it and hands me a permit. So far, so good. We meet Sandra Hirsch in the visitor parking lot, she greets us with a big smile and a cheerful „Moin, Moin“, a sure sign that we are now in Northern Germany and the gut feeling says we're in good hands. Ms. Hirsch drives an American car, Dodge Charger, with ample room for all. Do you have to drive a US-made car even in Germany when you work for a US company? "I also like Jim Beam", she responds, she clearly likes the car, it's that simple. The gatekeeper checks all permits and then opens. The Dodge surrounds us with a deep growl and begins moving. We are met with a surprisingly green landscape, the Elbe flows nearby at the Northern edge of the Chemistry Park and I can see the endless meadows at the river bank powdered with fresh snow. "We even have deer and wild boar here", says Sandra Hirsch. I don't see any, but I'll take her word for it. But where do the 1,200 Dow employees work? And

Andreas Kohler (left) and his colleague Sandra Hirsch assess a water sample extract. Before entering the Ecology Lab, clear and clearly visible signs alert you to workplace safety rules that must be followed at all times. Work safety is the highest priority at Dow, and not only in the laboratory, but throughout all production areas and the entire Chemistry Park Stade.



The GC Lab, part of the Ecology Laboratory within the Chemistry Park Stade, relies on GERSTEL solutions for much of its automation

where do they produce the three million tons of Dowanol, Methocel or MDI?

Where is the huge plant process equipment so typical for larger industrial chemistry sites where they produce and process allylchloride, chloroform and methylene chloride, sodium hydroxide and hydrochloric acid, propylene and...? The stacks and huge cylinders that require massive amounts of power, steam and compressed air? Where hydrogen, oxygen and nitrogen are produced or added to the chemical processes? Where are the pipes through which countless cubic meters of process and cooling water are pumped in and waste water discharged? No sign of Trinseo, Air Liquide or Evides, companies that generate added value here. "The area covers 550 hectares or almost 1400 acres", says Sandra Hirsch, "things are less visible in these wide-open spaces."

The Ecology Lab, where Sandra Hirsch, Andreas Köhler and Ute Schomacker work is located in the ground floor of a two story building at the edge of the Chemistry Park Stade, a few minutes by car from our meeting point. Since its inauguration in 1975, the lab has regularly been expanded and updated. The laboratory deals with environmental and water analysis in the Chemistry Park Stade, including monitoring workplace air, and ensuring compliance both with maximum allowable concentrations, as per German Occupational Safety & Health rules for workplace air, and with regulations for hazardous materials.

The lab monitors water to ensure compliance with legal requirements for water quality including adherence to maximum allowable concentrations laid down in the regulations issued by the Lower Saxony State Office for Water Economy, Coastal and Environmental Protection. Every year, the Ecology Lab team analyzes around 6,000 samples, making sure that maximum allowable concentrations of various chemicals are not exceeded. 60 % of the samples are water, mainly waste water. Air samples

that are required to be drawn regularly as part of routine monitoring protocols, or whenever a leak or too high workplace concentrations are suspected in the production area, make up 30 % of the samples. Soil samples are taken and analyzed, for example, whenever a new building is being planned or if doubts about earlier contamination need to be alleviated. Soil samples make up approximately 10 % of the annual sample load. "Added to all this, we get about 10 samples per month that are sent to us by the environmental protection agencies for comparative analysis. This is done in order to check the accuracy of our analysis results. The agencies can call anytime and draw samples anywhere in the entire Chemistry Park in order to perform their legally required controls", Sandra Hirsch explains. Laboratory routines are well established here, but it never gets boring for the three persons who run the lab.

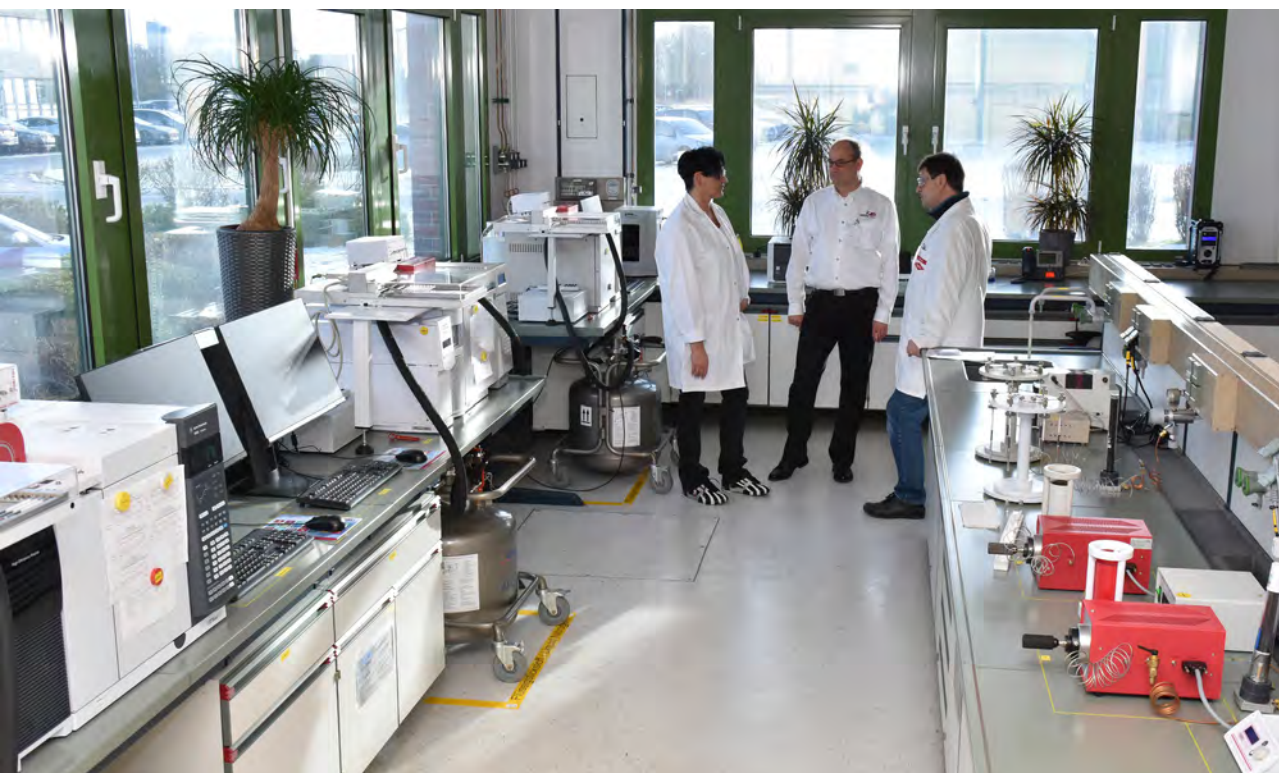
Occasionally there are even tasks outside the plant area, for example, when the Company Fire Brigade are cleaning up after a spill and need to know its chemical make-up. Or maybe the Fire Brigade from the town of Stade is handling an emergency or potential emergency, involving a road truck or a freight ship on the river transporting hazardous materials. "If they need chemical analysis, we are here to support them", says Andreas Köhler.

Occasionally, we have even had the Police Crime unit request an assessment from us as to the nature of substances found at the scene of a suspected suicide. The Ecology Laboratory takes on such tasks as technical support. Good to know: Technical analytical expertise can be found when needed. It is time for a tour of the laboratories. Sandra Hirsch and Andreas Köhler lead the way. The first stop for our small procession is the Thermal Desorption Laboratory.

The lab is remarkably orderly, it almost seems as if it had been cleaned up and made extra neat for our visit, but apparently that is the way they always work. Sandra Hirsch explains: "Searching takes a lot of time, right?"

I answer: "Right!". "And we don't have time", says Andreas Köhler. "And we don't need to either", adds Sandra Hirsch, "Because we know where everything is located". The method by which everything is organized originally came from Japan. It is termed the 5S or 5A method. The method specifies that everything must be in its place, must be clean, and must be ready for use. A clear sign that the 5A method is in use is the clear markings on the lab benches around important tools or instruments: Yellow lines marking areas on the lab benches, on the floor, on drawers, and on cabinet doors make it easy to locate the right tool or instrument and to handle it correctly. It reminds me of the areas designated for smokers in Railway Stations. An optically clearly distinguishable element can discipline people operating in the area, even though there are of course lots of yellow lines and signs in the lab.

On the lab bench across the aisle, two Tube Conditioners (TC 2), used to condition sorbent tubes, are standing ready within their yellow line frame. Right next to them are two Tube Standard Preparation Systems (GERSTEL TSPS), used to load sorbent tubes with standard mixtures. "For quite a while, we experimented with different sorbent materials, and today, using just one method, we are able to determine more than 50 analytes in the range from C10 to C40 with limits of determination in the single ppb range", Andreas Köhler says. "And we are far below the required limits of determination specified by the authorities", Sandra Köhler adds. The two colleagues clearly take great pride in their work and in reaching ambitious goals. To be ahead of regulations, and always stay ahead in the analytical race, drives them to great performance. This is also wise as a precaution: "We know from exper-



© Guido Deussing

The Thermal Desorption laboratory of Dow in Stade. Sandra Hirsch: „Colleagues from other sites are sending us their samples for analysis“.

Another color that dominates is GERSTEL magenta. Even a lay person would recognize that the lab has a high affinity towards GERSTEL products. On the lab benches along the windows, there are three complete GC/MS systems, each equipped with a GERSTEL Thermal Desorption System (TDS) with Autosampler (TDS A) mounted above a Cooled Injection System (CIS). "Apart from ensuring that the lab is well organized and neat, automation is the other way to improve efficiency", Sandra Hirsch remarks, "and this is especially important when you are not free to hire more people", as is the case in most labs. Compounds are detected using MSD and FID, connected to the GC column via a column effluent splitter.

rience", says Sandra Hirsch, "that rules and regulations change at short notice". Instead of waiting and reacting, the two experts are proactively meeting future challenges with steadily lower required limits of determination in order to be ready if these are enforced. "That approach has always served us well", says Andreas Köhler, "whenever stricter guidelines were enforced, we were prepared and ready". As to the method, he adds: "From volatile organic compounds (VOCs) with boiling points around -20 °C to semi-volatile compounds (SVOCs) with boiling points of 180 °C, we can generally determine them all using just one method". And the word is out within the worldwide Dow organization about the results the team has

achieved. The Ecology Lab in Stade is the center of excellence for thermal desorption analysis world-wide. Sandra Hirsch: “Colleagues from other sites are sending us their samples for analysis, that is quite a recognition”. But not just Occupational Safety and Hygiene samples are received from other Dow sites. Highly labor intensive AOX analyses, the results of which must be reported to authorities, are performed by the lab, which receives samples from sites in various countries. This is the work area of Ute Schomacker who has extensive knowledge in the field. By the way, Andreas Köhler discovered Thermal Desorption for his work before the year 2000. Sandra Hirsch, who today shares his enthusiasm, later joined the team.

When their “homemade” TD had reached its capacity limits, they decided to purchase a commercially made system: “We needed a system that enables accurate determination of the many compounds we must report with low limits of detection and without sample to sample carry-over” says Sandra Hirsch. Apart from that, Dow needed the potential to reach even much lower limits of determination as regulations grow stricter, coupled with efficient automation.

Early on, Andreas Köhler was interested in the GERSTEL TDS and in 2001 he contacted Michael Gröger, beginning what has today developed into a friendly professional partnership. The cooperation is not limited to the determination of VOCs in air. GERSTEL is also supplier of solutions for the analysis of water samples that make up the bulk of the samples in the Ecology Lab. In the GC lab, I count at least six GC systems with individual GERSTEL MultiPurpose Samplers (MPSs) mounted on top. Just like in the TD lab we just toured, the 5S yellow markings and GERSTEL Magenta stripes here accentuate the beige colored GC lab, which somehow exudes quality work. And as to the underlying quality of the analysis systems, Andreas

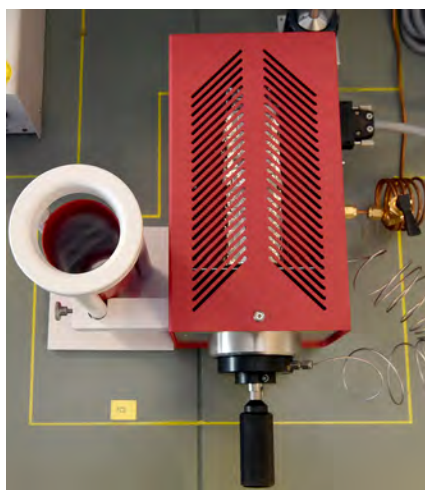
Köhler reports that in the field water analysis, the Ecology Lab has equally been faced with steadily tightened requirements, which they have been able to meet. Waste water with its high salt content and the various polar compounds that need to be determined had posed quite a challenge to the analysis experts. Many compounds can be determined with standard headspace GC analysis resulting in perfectly adequate results in terms of sensitivity and accuracy. Other compounds are less simple to determine, especially higher boiling compounds or those that are more water soluble. “The solution came in the form of GERSTEL’s HIT technology”, says Andreas Köhler. The acronym HIT signifies Hot Injection and Trapping. “The method ensures that higher boiling compounds are kept in the gas phase during sample introduction”, Andreas Köhler explains, “they don’t condense on the syringe needle and, more importantly, are not removed and lost from the inlet when the needle is retracted following the injection”.

In addition, analytes from multiple injections can be trapped and transferred together to the GC column for a single GC run. HIT has provided the Ecology Lab with a way to achieve better sensitivity, allowing them to stay ahead of the game for a long period of time. In the never-ending race for lower limits of determination, clearly, the team in the Ecology Lab takes great pleasure in being a step, or more exactly several steps, ahead of the authorities in meeting regulatory requirements. Sandra Hirsch: “one of our goals was to lower the limit of determination to 1 µg/L even though the requirements are 2 µg/L. We actually reached 0.2 µg/L”. And while others are struggling with standard headspace or liquid/liquid extraction techniques, the Ecology Lab staff has automated their analysis quite efficiently. This means more time is available for data handling, data interpretation and reporting – or for method development. “Or to ponder how much work an MPS WorkStation needs to perform in the lab”, says Sandra Hirsch, a friendly reminder that the tour through the lab is coming to an end and urgent work is waiting – I hear you.



Standing side by side as they have for many years, from right to left: Andreas Köhler and Sandra Hirsch from the Ecology Laboratory with GERSTEL Sales Manager Michael Gröger

© Guido Deusing



Structure promotes efficiency – also when it comes to operating the GERSTEL TC 2. The team in the Dow Ecology Lab keeps the lab orderly following the Japanese 5S method. The areas framed by clearly visible yellow lines are designated exclusively for one instrument. No other instrument or unrelated tool is allowed to be stored within this area.

© Guido Deusing