New calibration method for thermal desorption

■ Suitable sampling and measuring methods are required to determine organic compound contamination of interior and exterior air. Enrichment on thermally desorbable sorbents is a widely-used method, offering the advantage over solid-liquid extraction of allowing the entire sample to be injected for GC analysis all at once, which results in low detection limits. It also does not require the use of toxic solvents.

The calibration of the measuring process is particularly important. In co-operation with Reinhard Keller, Head of the Department at the Institute of Medical Microbiology and Hygiene at the Medical University of Lübeck, GERSTEL has designed a calibration unit taking six TDS tubes to make this stage faster and simpler.

Using a knurled nut, the TDS tubes can be fixed to the bottom of the unit via a Teflon ferrule, with six septumless sampling heads (SLHs) located on the top for contamination-free injection of the calibration solution. The entire unit is purged with carrier gas and the volumetric rate of flow can be regulated separately for each channel.

The pre-conditioned TDS tubes were clamped into the calibration unit. In his tests, Keller used analytes which are listed in VDI 4300, Sheet 6 for determining the overall concentration of volatile organic compounds and which also span a relatively broad boiling-point range (66 – 287°C). Between 10 ng and 2000 ng were selected for the concentrations to be examined so as to take account of all problematic concentrations occurring in interior spaces. The analytes were dissolved in methanol and doped at the rate of 1 µl was injected into the system to provide the proper loading of the compounds into the Tenax™.

The advantages of the process developed by Keller compared with conventional methods are obvious: less instrument equipment and time needed, easy handling and easy to integrate into laboratory practice. This means that an alternative to the established procedures, such as the use of test gases, now exists.

The GERSTEL Twister™ tested in practice

Waste-water analysis made easy

Differing TOC concentrations were found in the waste-water tanks of two plants producing under the same conditions, although the concentrations were virtually identical at the inlet to the tanks. In the troubleshooting process, the GERSTEL Twister proved to be the ideal means of identifying the constituents quickly and economically after thermal desorption, gas chromatography separation and mass-selective detection.

A chemical company in the German region of North Rhine-Westphalia manufactures a certain product in two different plants using an oxidation process, with the waste water accumulating fed into two separate tanks. Although the products and production conditions were identical, the waste water had differing TOC concentrations (Fig. 1), with tank 2 displaying higher values than tank 1. In order to be able to identify the cause quickly, an analytic method was required which would provide qualitative information about the constituent. The TOC concentration alone was not sufficient.

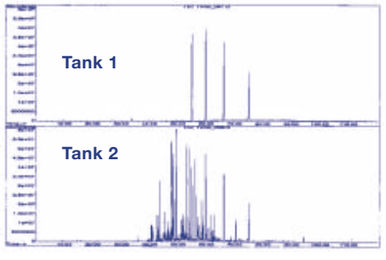


Figure 1
Waste-water tank TOC concentrations

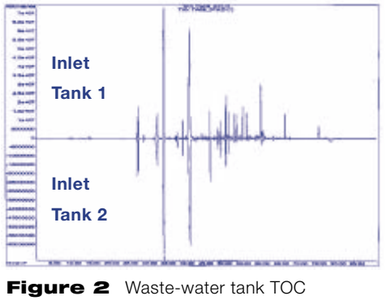


Figure 2 Waste-water tank TOC concentrations in the inlet pipes

Measuring method

The GERSTEL Twister was put directly into 20 ml of the samples taken from the inlet pipes and the waste-water tanks, with these then agitated for 30 minutes at a time. The constituents were sorbed in the Twister's polydimethylsiloxane (PDMS) coating. Analysis was then carried out by direct thermal desorption by placing the Twister in a GERSTEL TDS 2 in combination with the GERSTEL CIS 4, an Agilent GC 6890 and a GERSTEL MCS column switching system (column 1: 60 m RTX-1, ID 0.32 mm, FD 1.5 µm; column 2: 90 m Agilent VOC, ID 0.32, FD 1.0 µm) and mass-selective detection (Agilent MSD 5973).

Evaluation

While the chromatograms of the samples from inlet pipes 1 and 2 were virtually identical (Fig. 2), those of the tank samples displayed distinct differences (Fig. 1). The source of the discrepancy therefore had to be in the waste-water tank. As the Twister and the MSD allowed the components to be identified, potential causes of the additional contamination could be determined using available piping plans. This meant that there had to be at least one more inlet pipe to tank 2. This was located, after which a sample was taken and compared with the existing results. When the components found were added to tank 1, the chromatogram showed the same picture as in the analysis of the sample from tank 2 (Fig. 3 and 4). The cause of the additional waste-water contamination could then be established.

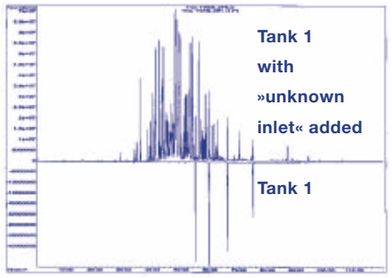


Figure 3
Waste water contaminant load with and without the components detected in tank 2

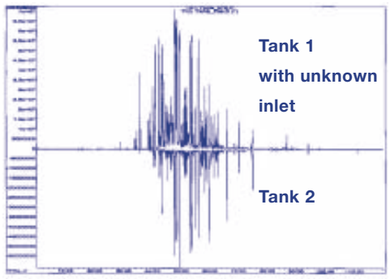


Figure 4
Comparison of contaminant load in tank 2 with unknown inlet

Result

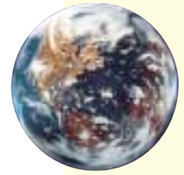
With the help of the GERSTEL Twister, it proved possible to implement a process which, compared with conventional sample preparation for GC analysis, enables an organic waster-water contaminant to be detected much more quickly and economically while retaining the same measuring sensitivity. The advantages of using the Twister: the costs were only one third of those normally incurred and the time required for the analysis was, after optimization, reduced to a quarter of that previously needed. Following identification with a mass-selective detector (MSD), the quantifying process can be carried out using a flame ionisation detector (FID). This possibility is currently being tested.

Analytic conditions

■ TDS:
Starting temperature: 5 °C
Heating rate: 60 °C/min
Final temperature: 240 °C
Final retention time:
5 minutes

■ Purge flow: 100 ml/min

■ CIS 4:
Glass evaporator tube with TENAX
splitless mode
Starting temperature:
– 100 °C
Heating rate: 12 °C/s
Final temperature: 240 °C
Final retention time:
20 minutes



ICB presents GERSTEL at Achema 2000

World's first through innovati

■ In the development of innovative an marketable products, the transfer of know how between industry and researc institutes can often be the key to developin technologies for the future. An exceller example of this is the co-operation betwee GERSTEL and the Institut für Chemo- un Biosensorik (ICB) in Münster, Germany i developing the TOPAS system: a badge shaped Tenax-based passive collecto combined with thermal desorption for direc transfer to GC analysis (see page 3).

While TOPAS has left the experiment stage and is currently undergoing practic testing, everyone is now talking about ye another new innovation resulting fro GERSTEL's co-operation with the ICB: th Automated Speciation Analyser (ASA), new type of analytical instrument whic thanks to its special column technology an an element-selective detector, provides fo much faster GC analysis than hithert facilitated by conventional systems. As Ra Bremer, technical director at GERSTEL says: »The ASA is designed to enable high toxic metallo-organic compounds to b detected for the first time in a manner tha is fast, reliable, sensitive and extreme economical.«

Bringing these attributes together in a sin gle product was an analytical challenge. » took a huge amount of technical and tim resources,« comments Dr. Wolfgan Buscher, ICB's analyst. With the ASA organic mercury compounds, for example can be detected five times more quick than with conventional methods. Th detection limit is 2 picogrammes in absolu te terms. The analyst is convinced tha future prospects are bright.

Further information is available fro ICB Münster: w.buscher@izb-online.de.