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Environmental Applications of Trap & Thermal Desorption in Combination with a Portable GC

W. Muenchmeyer, A. Walte
WMA Airsense Analysentechnik GmbH
email: info@airsense.com http://www.airsense.com
T:+49.385.3993.280 F:+49.385.3993.281

Applications by: G. Stani, A. Miliazza
SRA Instruments Italia S.R.l
email: info@srainstruments.com
http://www.srainstruments.com

Overview

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Portable and fast instruments for gas chromatography are increasingly used for on-site or in-line analytical tasks. In many demanding applications, improved sensitivity and/or selectivity is required.
By coupling a specialized and fast Trap/Thermal desorption technique [1] to a micro gas chromatograph detection limits required in environmental and industrial applications are reached.
The investigation of different operating parameters show the capabilities of the solution.

Introduction

2

A Micro-GC (MGC) equipped with a short column and a micromachined thermal conductivity detector (TCD) provides the capability of quick measurements with good analytical properties on-site or in-line.
However, in many applications, detection limits at low ppb levels are required.
Using the well known Trap/Thermal Desorption Technique for gaseous compounds detection limits in the required concentration range are possible. Also an increase of resolution capabilities can be reached by the extraction of disturbing compounds from the analysis during the sampling on adsorbent materials.
In order to maintain the application characteristics of a Micro-GC, a number of features must be fulfilled by the instrument design. Beside the analytical properties the size as well as the power consumption and the capability of continuous operation must be considered.

Methods

3

The instrument, named µ-TD® includes a very light thermal desorber for heating the adsorbent material contained in a standard glass tubing. With this thermal construction, the temperature of the adsorbent bed can be cycled at a high rate and the same system can include the sampling step and the thermal desorption within one cycle. A cleaning step within the cycle ensures the adsorbent to be clean after a cycle (<0.1 ‰) so that continuous operation with one tube is possible. In fig. 1 the schematic set up of the complete portable system is indicated

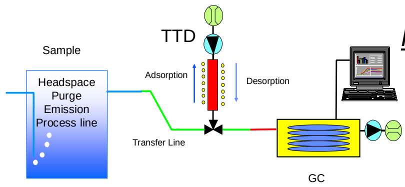


Fig 1: Principle of set up

Fig 2: Flow scheme of µTD

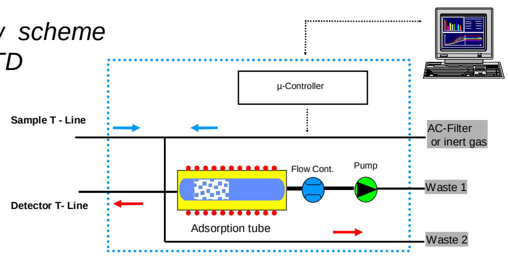


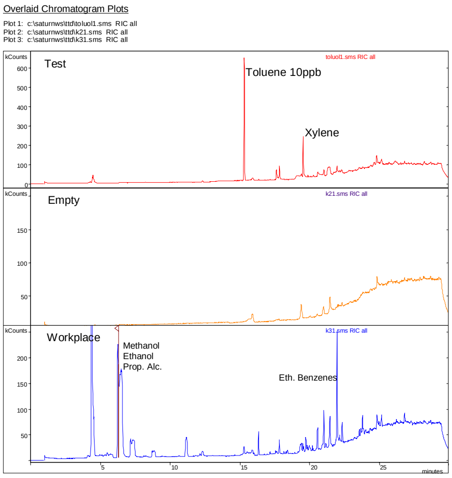
Fig. 2 shows the flow diagram of the Trap/Thermal Desorption system. Several ports for clean and waste gas are switched during a cycle in order to achieve an application flexible design. The instrument performs the following steps of analysis:

1. Standby: no flow over tube, clean gas flow to detector possible
2. Post-Sampling: clean air to rinse tube
3. Sampling: sampling flow through adsorbent tube, clean gas flow to detector possible
4. Desorption: adsorption tube heated , clean gas flow to detector possible
5. Injection: reverse flow from tube to detector
6. Cleaning& Cooling: tube rinsed at high temperature, clean gas flow to detector possible

Results

5 The Trap/Thermal Desorption unit has been connected to a lab GC/MS (Varian 3800/Saturn 2000). For sampling, different standard test gases have been diluted in sample bags. A selection of GC-runs is displayed in fig. 3. Toluene at 10 ppb and Xylene at 1 ppb has been sampled for 10 minutes at a flow rate of 300ml/min (Tenax TA). The signal from these substances is far above the S/N detection limit.

Fig 3: *Examples of laboratory measurements*



A following run with an empty sample shows that the amount of sample remaining in the system is very small ($<10^{-4}$) so that a tube can be used for the next sampling. A sample from a workplace environment (5L vol. on Tenax/AC) shows comparative sensitivities with some peak broadening at high volatile compounds and a good separation with MVOC. Compounds of a broad range of volatility can be sampled with a combination of different adsorbent materials.

Clean air from active charcoal filtering was used as cleaning and injection gas when attaching the Trap/Thermal Desorption Unit with a Micro GC (fig. 4). A start button puts the instrument into operation for one cycle or endless repetition of cycles. For the injection, a start signal is released to the Micro GC and the sample is transferred to the GC. A heated transfer line (Silcosteel) ensures that no substance loss occurs in the analytical pathways.

Fig.4: *TTD unit Attached To Agilent Micro-GC*

6 With this simple set up a good sensitivity in the range of 10ppb and a good reproducibility (rsd<1%) is achieved for aromatic compounds. For simple tasks, the use of filtered air for injection is sufficient. Further avoidance of artifacts can be achieved using the GC carrier gas for injection.

Some properties of the Trap/Thermal Desorption Instrument are listed in Table 1.

The trap can be used for monitoring applications, but also for single thermal desorptions. Fig 5 shows the instrument alone and the easy changing of the adsorbent tubes.

Adsorbent	Tenax TA, 125mg, Tenax TA/ AC, others available
Sampling Flow	Adjustable 50..400ml/min
Desorption Temp.	Adjustable, ...300°C
Sampling & Transfer	Heated tubes stainless steel / silcosteel 50...150°C
Cycle time	Typical 6 to 8 min
Interface	RS 232 (parameter download), TTL, Relay
Power	110 ... 230 VAC, 12VDC (opt.), max. 80 W
Size	230 x 285 x 68 mm , 2,3 kg

Table 1: *Technical properties of µTD*

Fig.5: *Specialized TTD unit (µTD) optimized for operation with a Micro-GC*

Conclusions

7
- The use of Trap/Thermal Desorption increases the sensitivity of a Micro-GC
- A design for a small size instrument with low power consumption and autonomous operation offers a specialized solution for Micro-GC Applications.

[1] A. Walte, W.Muenchmeyer "Enrichment device for Electronic Noses", Poster 1693P, Pittcon 1999