

PPB-Level Organic Gas Detection with Surface Acoustic Wave Based Sensor Systems

M. Rapp, K. H. Lubert, F. Bender, A. Voigt
Forschungszentrum Karlsruhe GmbH
Institut für Instrumentelle Analytik
Postfach 3640, 76021 Karlsruhe, Germany
E-mail: michael.rapp@ifia.fzk.de

The use of polymer coated surface acoustic wave (SAW) sensor arrays as the key element for analytical sensor systems is a very promising technique for organic gas detection for process control and electronic nose applications. In the past years, we have shown the potential of this technique by using self developed SAW sensor systems basing on arrays of single commercially available SAW resonators in TO39 cases. Nevertheless, such sensor systems suffer some drawbacks such as unwanted dead volume, unexpected long sensor response times of minutes instead of seconds, memory effects and high production costs. Our solution is a novel concept to achieve further miniaturization of the SAW sensor array with a dramatic reduction of sampling volume and inner surface area to minimize response time and unwanted adsorption phenomena. The SAW micro array is directly integrated into the electronic setup leading to small detection tools that are fast, portable and cheap. Contacts of interchangeable SAW sensors are made by a new capacitive coupling technique using contact pads on the circuit board facing corresponding pads on the SAW sensors. Though, no bond wires are required but each sensor is still separately interchangeable! Gas sampling is achieved via two milled channels on the circuit board underneath 4 sensors in line, respectively. This results in a sampling volume of only 50 μ l! Each channel is totally covered with a gold over-layer obtained via standard chemistry. Sampling gas come into contact only with gold or the polymeric sensing layers on the SAW sensors, resulting in very fast response times of 2-4 seconds!

Optionally, the sensitivity of this array can be enhanced by using a self developed pre-concentration unit (trap). The unit aspirates gas at high flow rate through a tube filled with a material of high absorption capability. Periodically, this material is heated and the thermally desorbed vapor is pumped through the sensor array at low flow rate. The thermal capacity is kept very low in order to get short cycle times of a few minutes combined with high pre-concentration factors (up to several thousand) due to large sampling volumes. The devices are exposed to the pre-concentrated sample for only a few seconds, necessitating the rapid sensor response of the SAW sensor micro array. In addition to an increase in sensitivity, this approach offers several other important advantages:

1. Influence of major interfering components (such as humidity) can be suppressed by an adequate selection of adsorbent material(s).
2. Enhancement of Sensitivity: The pre-concentration factors of 1000 or more can be achieved for the most important analytes, the concentration of these analytes in unchanged indoor air can be neglected in comparison to the pre-concentrated sample.
3. Rapid fluctuations in analyte concentration – as they occur e.g. in close proximity of a leak – are effectively suppressed by continually collecting the vapor in the pre-concentration unit, making pattern recognition more reproducible.
4. Long- and medium term drifts are no longer a matter of concern! Independent from the real time of collecting time (range of 4 – 60 min), the time of measurement is constant and short, only 10 seconds.
5. No reference gas is required: The system can use the not-enriched analyte gas as zero gas. Thus, its signal can serve as a base line versus the subsequent signal of the pre-concentrated sample which is expected to be significantly higher.

Our experiments show that this new sensor concept combines highly reliable analyte identification with the capability to detect concentrations in the low ppb-range. Fig. 2 shows the complete systems with sensors system and trap. Its actual limits of detection (LOD) of various gases using a trap with tenax as absorbent resin are shown in fig. 3. The properties of the resin is mirrored in the LOD's: For unpolar gases the values are in the ppb-range, for polar gases they are in the ppm range. The behavior can be inverted if polar resins would be used. With other sorbent resins like "Carboxen 1000" provided from Supelco/Aldrich, a nano-porous carbon material, even the enrichment of gases with a low molecular weight was successful such as refrigerants or ethylene oxide. This presentation will show some actual applications in the field of work place and clean room monitoring, forensic applications (detection of drugs and explosives) and a new type of early state fire detection. The latter is now possible in a really early stage: Already the overheating of electrical wire isolation or circuit board above their nominal working temperature (usually 120°C–150°C) can be detected significantly!

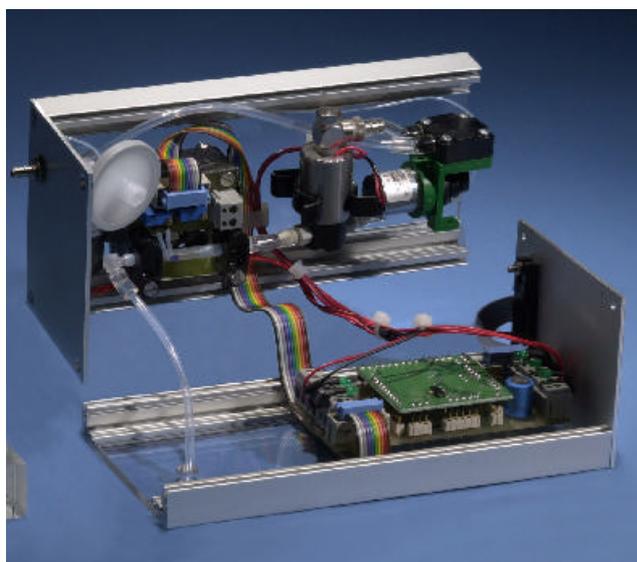


Fig. 1:
Trap module for analyte gas enrichment with electronic controller, pumps and valves. The unit is able to perform complete sample cycles within



Fig. 2:
Complete SAW sensor system with trap unit as a prototype for a hand-held instrument with a size of 17 x 17 x 8 cm³.

Analyte	Molar mass [g/mol]	Boiling point [°C]	Cycle time [s]	Sensitivity [Hz/ppm]	Detection limit [ppb]
Naphthaline	128.2	218	420	47,000	11
n-Decane	142.3	174	240	34,000	15
Styrene	104.1	145	240	30,000	16.5
Xylene	106.2	140	240	16,000	31
Octamethylcyclotetrasiloxane	296.6	176	240	14,000	35
Ethylbenzene	106.2	136	240	13,000	38
n-Octane	114.2	126	240	11,000	46
Toluene	92.1	111	240	5,800	86
Tetrachlorethene	165.8	121	150	5,200	96
Benzene	78.1	80	600	1,200	410
n-Heptane	100.2	98	140	907	550
Tetrachlormethane	153.8	77	600	412	1,200
n-Hexane	86.2	69	240	214	2,300
Acetone	58.1	56	240	79	6,400

Fig. 3:
Actual limits of detection using tenax as absorbent resin for the preconcentration unit