Defiant Technologies Inc
6814A Academy ParkwayW NE
Albuquerque, NM 87109
FAX:505-341-9788
www.defiant-tech.com

Gas Sensor Operating Principles

1 INTRODUCTION

The Canary^{TM} series of sensor systems capture a gas sample in a chemically-selective porous media, then thermally desorb the sample in a sharp, concentrated pulse. In the Canary-One^{TM}, the sample pulse moves to a detector where it is momentarily captured in a polymer and weighed. This process is illustrated schematically in Figure 1. The selectivity of the porous media on the collector and the coating on the detector offers the ability to identify the target gas sample while disregarding interferants, but it has limited specificity.

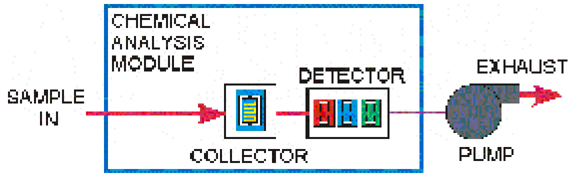

Figure 1. Canary-One^{TM}

For greater specificity, the Canary-Two^{TM} incorporates a micro-gas chromatograph (GC) column as shown schematically in Figure 2. A sample enters the GC in a sharp pulse, then separates into individual constituents as a result of interactions with a coating, called a stationary phase, on the column walls. As the constituents emerge from the column, they temporarily collect in the detector. The time between sample injection and emergence of the constituents from the column is used to identify the analyte. The measured weight of the sample is related to the concentration of the analyte in the gas sample.

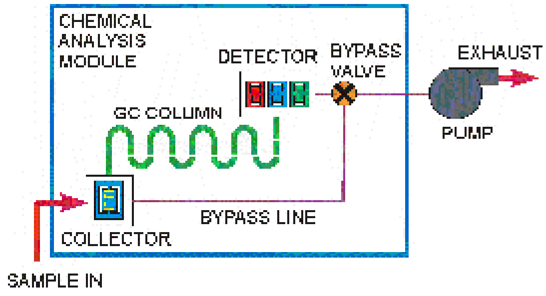

Figure 2. Canary-Two^{TM}

Although the Canary-Two^{TM} provides a more detailed assessment of the concentration and composition of a gas sample, the two minute analysis time may be unacceptable for survey and screening situations. Thus, the Canary-Three^{TM} incorporates dual detectors. The sample path for the first bypasses the GC to provide a quick survey of the surroundings, while the sample path for the second incorporates a GC and may be switched in to provide a more complete analysis when needed. This is illustrated in Figure 3.

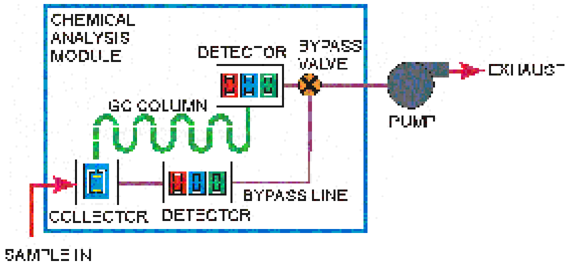

Figure 3. Canary-Three^{TM}

Specific performances and operational characteristics of each device are provided in Defiant's Product Brief.

2 SYSTEM COMPONENTS

2.1 Collectors / Preconcentrators

The first stage of the analysis system is a sample collector, also called a preconcentrator, where analyte samples are trapped in a coating. Upon heating, the trapped sample thermally desorbs and proceeds to the GC and detector. Defiant selects among three types of preconcentrators based on the vapor pressure of the analyte and concentrations of interest. For high vapor pressure compounds, such as volatile organic compounds, the tortuous path preconcentrator shown in Figure 4 is used. The finned silicon structure provides a high surface area for sorbtive coatings, yet the structure has sufficiently low mass and high thermal conductivity to permit rapid heating during the sample desorption process.

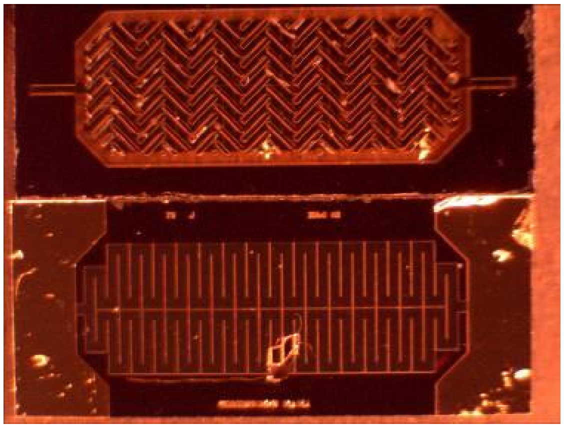

Figure 4. Tortuous path preconcentrator

For low-vapor pressure compounds, such as pesticides and explosives, a micro-plate suspended on two struts is used (see Figure 5). The lower mass and enhanced thermal isolation of pivot plate preconcentrator allow faster heating for a sharper injection of samples into the GC column. Preconcentrator coatings are more efficient at capturing the low vapor pressure analytes, so even with the lower surface area it collects sufficient analyte for analysis and the sharper injection creates sharper peaks in chromatograms from the 1-2 meter length columns used in Defiant's systems.

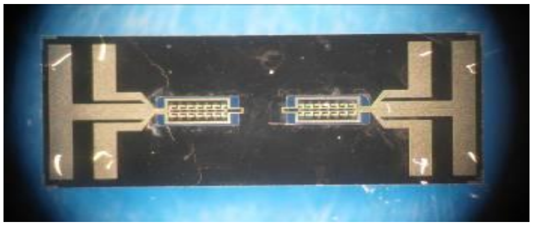

Figure 5. Pivot plate preconcentrator

A third option offered for the preconcentrator is shown in Figure 6. The concentric ring structure offers a higher surface area than the pivot plate and yet has a lower mass than the tortuous path. It is a cross-over design that operates over a broad cross-section of analytes. The flow-through design of this preconcentrator also allows a more direct injection into the GC columns without the need for turns in the flow path.

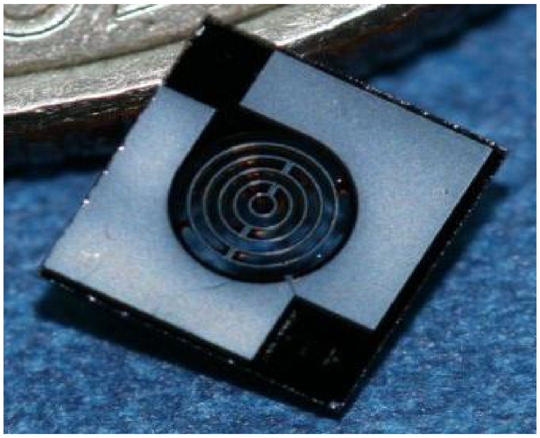

Figure 6. Concentric ring preconcentrator

Coatings on the preconcentrator must be able to collect a vapor sample and hold onto it throughout the collection cycle but, when heated, to rapidly release the sample in a pulse that is sharp relative a changing background concentration. The coating acts more like a sponge than a bucket – target analytes will diffuse into the coating until equilibrium conditions are established with the gas stream. Defiant typically uses solgel coatings. The high collection capacity of solgels make them suitable for a wide range of sample concentrations – they effectively scavenge the air for analytes at low concentrations, and conversely they can collect large amounts of analyte at high concentrations before becoming saturated. The solgels are modified to improve collection of different compounds. These modifications are achieved through additives and processing conditions during the curing process. Solgels can be selectively tailored for low-vapor pressure compounds (such as pesticides or explosives) or high-vapor pressure compounds (such as volatile organic compounds).

2.2 Micro Gas Chromatograph Column

Defiant uses the micro-GC column illustrated in Figure 7. The column consists of an array of holes formed in a nickel coupon, with caps on either end of the array that route the flow into a serpentine path. The center array coupon and the end caps are formed in a LIGA process, so the column is referred to as a LIGA-GC. A one-meter LIGA-GC is approximately the size of a dime, and a 10-meter column is slightly smaller than a sugar cube. As in commercial columns, a stationary phase coating is applied to the inner wall of the column to aid in gas separation. The compact size of this GC column simplifies temperature control and allows the GC to be rapidly heated for faster chromatography.

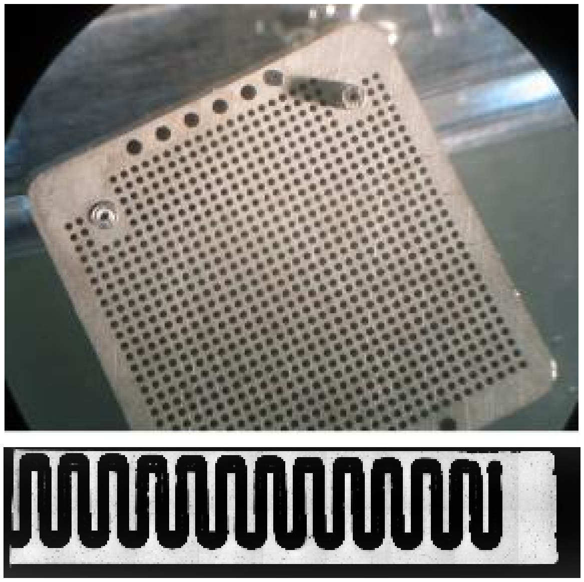

Figure 7. Micro-GC column showing top and cutaway views

2.3 SAW Microbalance Detectors

A surface acoustic wave (SAW) microbalance is a resonator that operates on a principle similar to the quartz crystals in most of today’s clocks. An alternating voltage in a primary pair of conductors on a quartz surface establishes a compression wave that moves across the surface. A second pair of conductors is in the path of this wave, and, as the wave passes, a current is developed in the secondary conductors. Technically this is referred to as a delay line SAW and any mass that accumulates on the quartz surface in the wave path will affect the delay in time between the launched wave from the primary conductors and the detected wave on the secondary conductors. By measuring this time delay, it is possible to measure mass down to picogram levels.

Mass is introduced onto the surface by absorption into a polymer coating on the quartz. A GC column discharges near the surface of the SAW and analytes are absorbed by the coating. The polymers release the analytes when clean air flushes over the surface. By selecting coatings with targeted analyte preferences, a unique signature is developed for every analyte.

Like the LIGA-GC, the SAW is roughly the size of a dime. One of the SAWs used by Defiant is shown in Figure 8. This particular design has two delay lines for measuring mass, and one reference delay line to compensate for temperature changes in the quartz. The mass sensing area is approximately 300 microns by 800 microns. The two rectangular blocks shown in Figure 8 are application specific integrated circuits (ASICs) that use a feedback amplifier to establish the wave in the quartz and a Gilbert cell mixer to measure the time delay between the launched and detected wave.

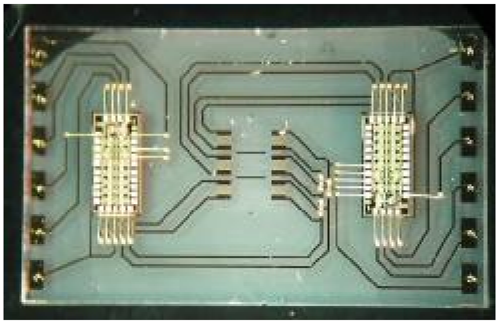

Figure 8. SAW microbalance detector

3 SYSTEM INTEGRATION

The three basic components of the gas analysis system are individually micro-fabricated and then integrated in a manifold that makes the appropriate electrical and gas connections. An integrated Canary-Three™ system, with preconcentrator, GC, a SAW microbalance, and a second SAW in the bypass line, is shown in Figure 9. A 2-way valve alters flow through the system from the bypass path used in collection, to the analysis path through the GC. The individual manifolds for each component are connected using inert tubing and epoxy, and then the assembly is screwed to a circuit board. Spring loaded probes embedded in the manifold modules make the electrical connections to the circuit card. By making relatively simple changes in the circuit card, a variety of manifold configurations can be constructed to suit different analysis needs.

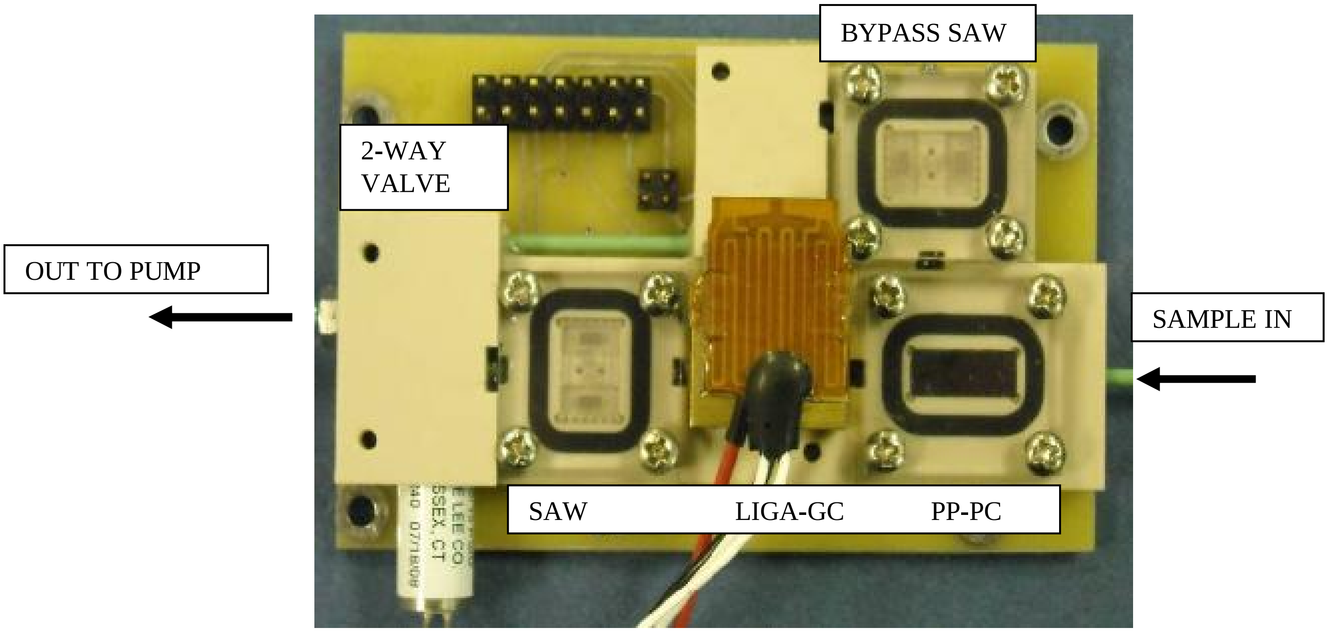

Figure 9. Integrated Canary-Three™ gas module.