DEVELOPMENT OF ON-LINE INSTRUMENTS FOR MONITORING OF METHANE AND NON METHANIC TOTAL HYDROCARBONS AT PPB AND PPM LEVELS WITHOUT MATRIX EFFECTS

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Monitoring of methane and Non-Methane Total Hydrocarbons (NMTHC) is an important tool for process monitoring. In the microelectronics industry, the silicon substrates (wafers) are stored in containers, named FOUP (Front Opening Unified Pod) made in polymers (PC, PEEK, COP, PEI). Such materials are able to adsorb the Volatile Molecular Contaminants (VMC) and subsequently outgas these ones in presence of wafers, leading then to defective wafers. Depending on the process, the composition of the gas in the FOUP can be very different. The amount of VMC can vary from ppb to tens of ppm while the matrix gas can be air, N2 or Argon. Most of the instruments which measure NMTHC are very sensitive to the matrix composition. Therefore, there is a need for an instrument which can measure methane and NMTHC without matrix effects. Ideally, the cycle time of the instrument should be less than 3 minutes and sensitive at ppb level.

Instrument configuration

Turnkey solution for Total VOC monitoring

An automatic isothermal gas chromatograph with a 250 µl loop injection, equipped with a Flame Ionization Detector and two chromatographic column has been developed. One column (short column) was dedicated to the separation of methane from the matrix gas and the second one (long column) to prevent the NMTHC to enter the short column. A 10-port diaphragm valve allows to perform a backflush during the analysis (Figure 1). The sampling flow was set to 25 ml/min.

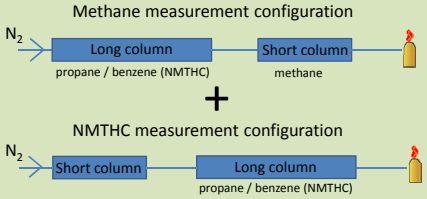
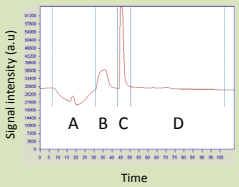


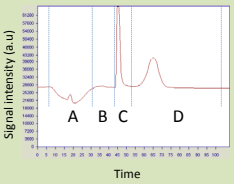
Figure 1: Gas flow depending on the 10-port valve position

Results

N2 and O2 effects on FID detectors



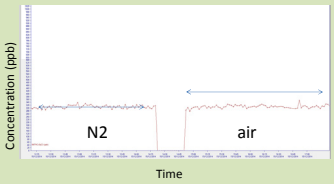
GC-FID chromatogram of 2 ppm methane in air



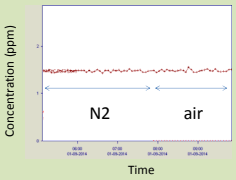
GC-FID chromatogram of 1,5 ppm Benzene in N2

A: O_2 and N_2 from matrix
B: Methane
C: Valve actuation
D: NMTHC

Tests with air as sample matrix



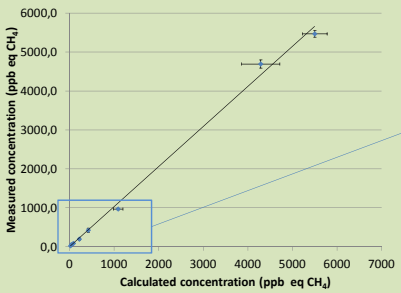
GC-FID trend of 300 ppb benzene in air and N2

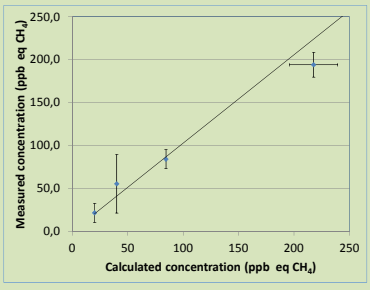


GC-FID trend of 1,5 ppm benzene in air and N2

Cycle time: 2 minutes

Linearity





High concentration:
Injected:
- 5500 ± 110 ppb (eq CH4)

Measured:
- 5469 ± 81 ppb (eq CH4)

Low concentration:
Injected:
- 20 ppb (eq CH4)

Measured:
- 21 ± 11 ppb (eq CH4)

LDL = 20 ppb ($eqCH_4$)

The results show that the special configuration of the analytical system allows the complete separation of the matrix gases from methane and NMTHC. Therefore the instrument does not need specific calibrations for different matrix. The instrument is very linear and sensitive from low ppb up to 20 ppm with the same amplification.