



Schematic description of negative temperature gradient band-narrowing procedure

Contacts:

Ing. Jan Beránek
Czech Metrology Institute
Department of Primary Metrology of Gas Mixture and Certification of Reference Materials
CZ, 102 00 Prague
E-Mail: jberanek@cmi.cz

Dr. Kaj Nyholm (project coordinator)
VTT MIKES, Tekniikantie 1, P.O. Box 9,
FI-02151 Espoo
E-Mail: kaj.nyholm@VTT.fi

Partners and Associates:

VTT MIKES, Finland (Coordination)
CMI, Czech Republic
INRIM, Italy
NPL, United Kingdom
PTB, Germany
VSL, the Netherlands
HC Photonics Corporation, Taiwan

Method based on the use of negative temperature gradient for off-line AMC monitoring

In the framework of the EMRP project MetAMC, CMI developed and tested a method based on the use of a negative temperature gradient for off-line analysis of airborne molecular contamination (AMC) using gas chromatography (GC). The technique enhances the signal-to-noise ratio of GC analysis.

Technical description

AMC including benzene and toluene at a few ppb at most must be measured reliably and accurately. However, a GC analysis cannot be done without application of pre-concentration and sample treatment techniques. Desorption of the compounds from adsorbents will unfortunately broaden peaks. Before injection onto the capillary column, it is thus necessary to focus the analyte band to reach sharp peaks and to have a high signal-to-noise ratio. Smart focusing means to cool a selected area of the column. Cooling causes the analyte to spend more time in the stationary phase, which slows it down. The leading edge of the sample band will travel slower compared to trailing edge.

For quantification, it is possible to use the GC technique with an FID (flame ionization detector). FID instead of a mass spectrometer has the advantage of almost equal carbon atom response for all hydrocarbons except ethyne. Consequently, even not-calibrated compounds can be quantified.

The foundation of the system is an enriching trap and a cryo-focusing column. The temperature of the enriching trap and cryo-focussing column is controlled by dipping into liquid nitrogen and electric heating while pulling the columns from the liquid nitrogen. The enriching trap is made as a "U" profile from ss-1/8" tubing and it is filled with Carbopack BHT. The cryo-focusing column in "U" shape is made of Ulti-Metal WCOT CP7120. Subsequently, the gas sample is analysed on GC with FID.

Advantages of the negative temperature gradient method

- better resolution (signal-to-noise ratio)
- possibility to use the GC technique with FID

Economic significance

AMC affect the product yield and inhibit the advance to smaller scale manufacturing techniques. The effective implementation of AMC monitoring equipment can enable more cost efficient fabrication and fabrication of new, innovative products and thus increase the overall competitiveness.

Acknowledgement

This work was funded by the European Metrology Research Programme (EMRP). The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union.