Progress in the development of a GC-APPI source with femto-gram sensitivity

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Introduction

ASMS 2013:
At the ASMS conference 2013 we introduced a new approach for a GC-APPI interface attached to a high resolution Exactor™ Orbitrap mass spectrometer [1]. The principal idea of the source design was rather simple: A tightly sealed ionization volume and a chemically and photo-physically inert matrix. In combination with the well defined sample injection via the GC this concept is in full accord with the necessary prerequisites for pure and efficient direct photoionization.

ASMS 2014:
Details of a significantly improved GC-APPI interface are presented.

Methods

mass spectrometer
Exactor™ Orbitrap, Thermo Scientific
 sampling rate: 10 Hz
 resolution: 10 000
 scan range: 50 – 1 000 m/z

custom ion source
nourin with low ppb/pm impurity level
 flat gasket sealing (Sigralflex®. A.W. Schulte, Geesthacht, Germany)
 400 W heater and power supply
 ion source material Invar36
 cone coating: electrochemical gold layer or PVD double layer of Al and MgF 2
 Syagen Kr RF lamp with power supply
 Omega® CC-High Temperature Cement
 inorganic coating: Ipsalux (Indestructible Paint, Ltd.)

gas supply
nitrogen with low ppb/pm impurity level
 compressed gas cylinder
 Vici Metronics 30 gauge purifier
 mass flow controller (Bronkhorst)

gas chromatograph
GC 450 series, Thermo Scientific
 column: TR-5 (93.5 m x 0.25 mm ID x 0.1 µ)
 GC transfer line: 335°C
 Helium (99.999 %) with 1.5 ml/min

samples
EPA 8270 LCS Mix 1, Supelco
 dilutions: 50 µg/l – 1 mg/l

CFD simulations
Autodesk Simulation CFD

Conclusions

Improvements:
O-rings replaced by Sigralflex® flat gaskets
cemented MgF 2 window
ion source enclosure made of Invar36
conical ionization volume and asymmetrical make-up gas inlet maintain a vortex flow pattern
GC-flow injection into vortex core
carefully balanced system of irradiation time, convectional and diffusive peak broadening, and radiation overlap with the ion current
 careful surface finish of the ion source cone

current performance:
24.7°C heating of the entire setup at 325°C for several weeks
no background except from column bleeding starting at around 280°C
peak width down to 0.6 s (FWHM)
lower limit of detection in the fg range

see also:
A.C. Peterson et al. 62nd ASMS Conf., Baltimore, MD, 2014, MP 364.10

Literature

Acknowledgement
Thermo Scientific is acknowledged for supplying the Exactor, the GC and the GC column.

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