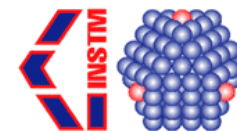
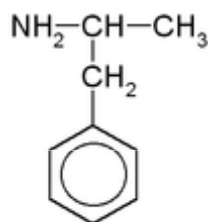
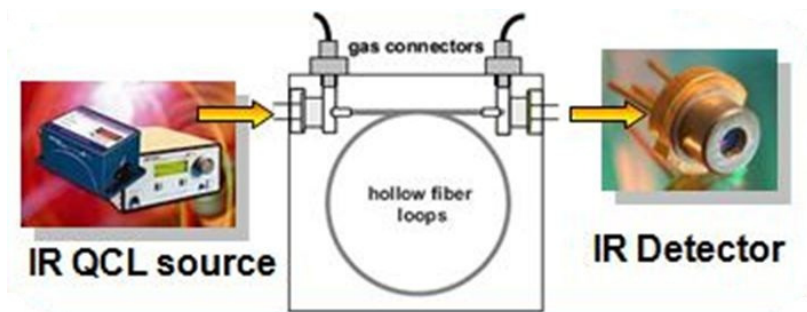


*FP7 Project **'DIRAC'**:
Detection and identification of illicit drugs by
IR absorption spectroscopy and gas
chromatography*

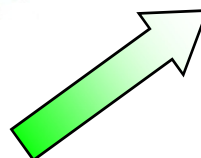
*N. Liberatore, D. Luciani, S.Mengali, R. Viola (CREO);
G.Cardinali, S. Zampolli, I. Elmi, and A. Poggi (CNR IMM);
E. Dalcanale, E. Biavardi and D. Menozzi (INSTM)*



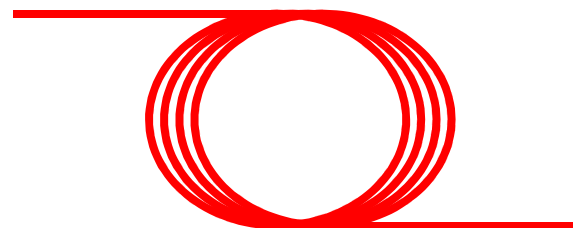
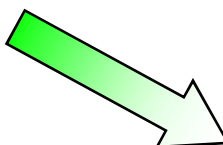
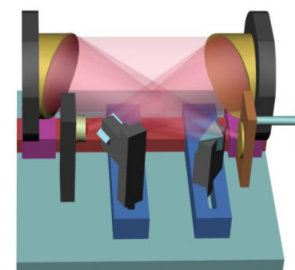
Introduction: The IR Hollow Fiber Advantage



3 ng of amphetamine
correspond to a
concentration of:



... **2 ppb** when vaporized in a
'standard' multi-pass cell (vol. $\approx 0,1$ l)

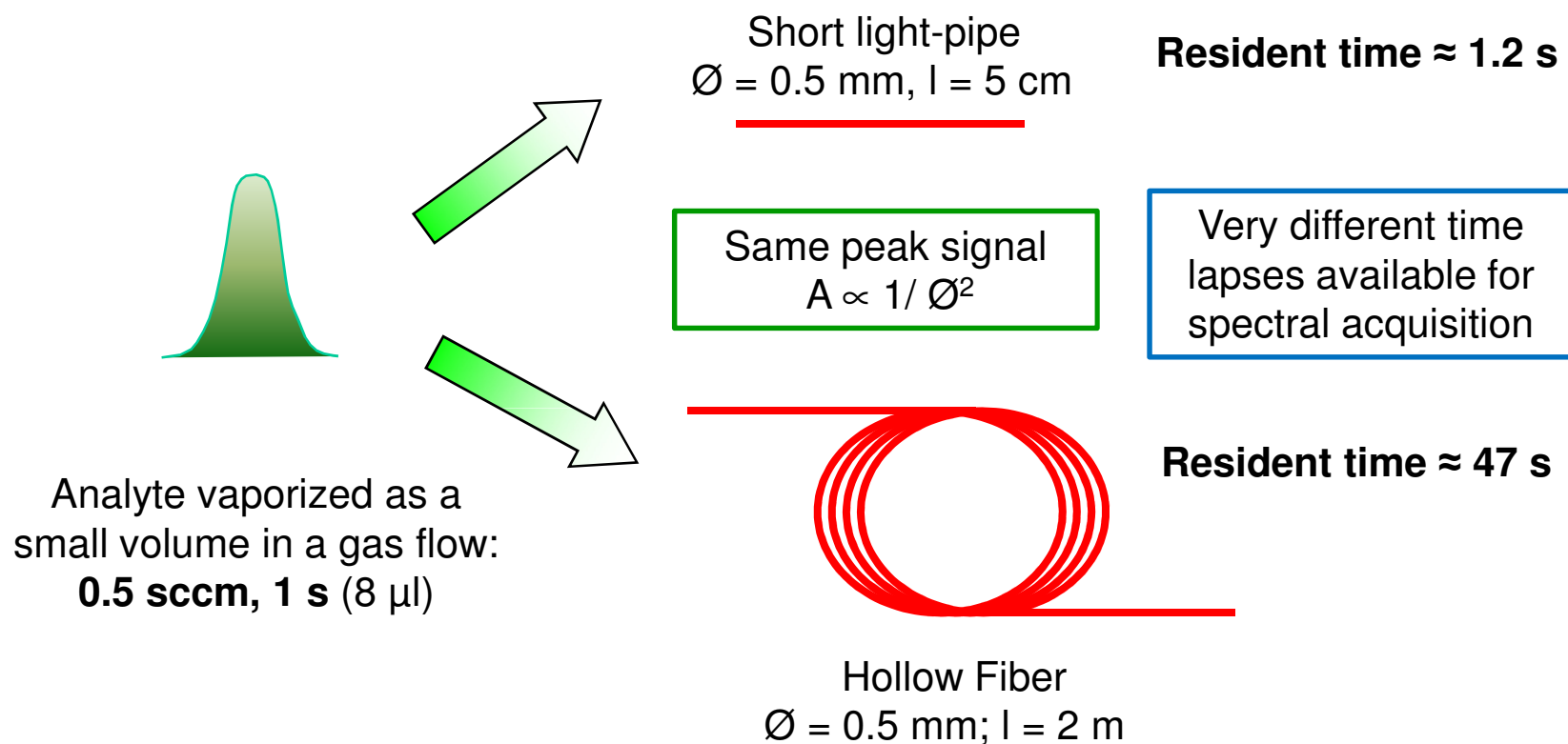


... **1ppm** when vaporized in a
hollow fiber $\varnothing = 0.5$ mm, $l = 1$ m (vol. $\approx 2 \times 10^{-4}$ l)

*HF is an optical cell of very simple,
cost effective, miniaturized design.*

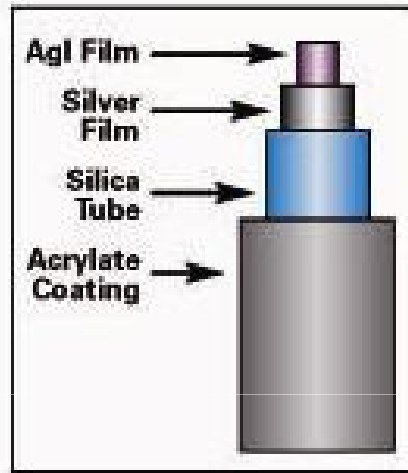
*If we can neglect optical losses, HF
provides higher sensitivity and
lower LoD than standard cells of
same optical path.*

Hollow Fibers vs Light Pipes



Optical Losses in the fiber

HF from
Polymicro Technologies



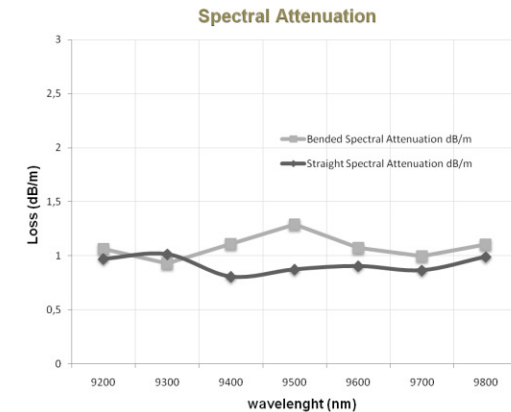
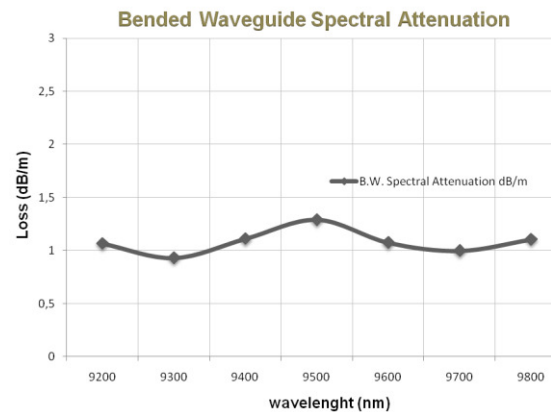
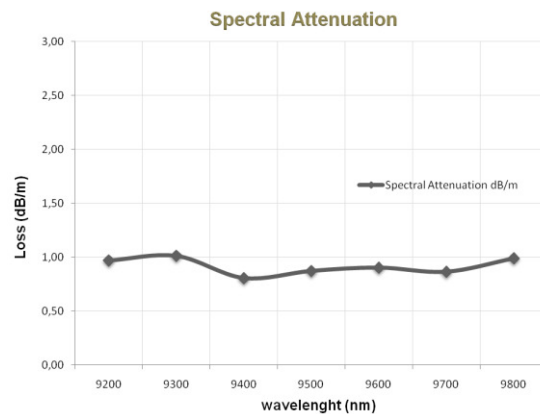
Measured straight losses ≤ 1 DB/m

Measured bend losses ($r = 45$ cm) ≈ 1 DB/m

Coupling losses $\leq 10\%$ / coupling

Temperature degradation: measured negligible at 130 °C over several days; negligible at 150 °C according to manufacturer

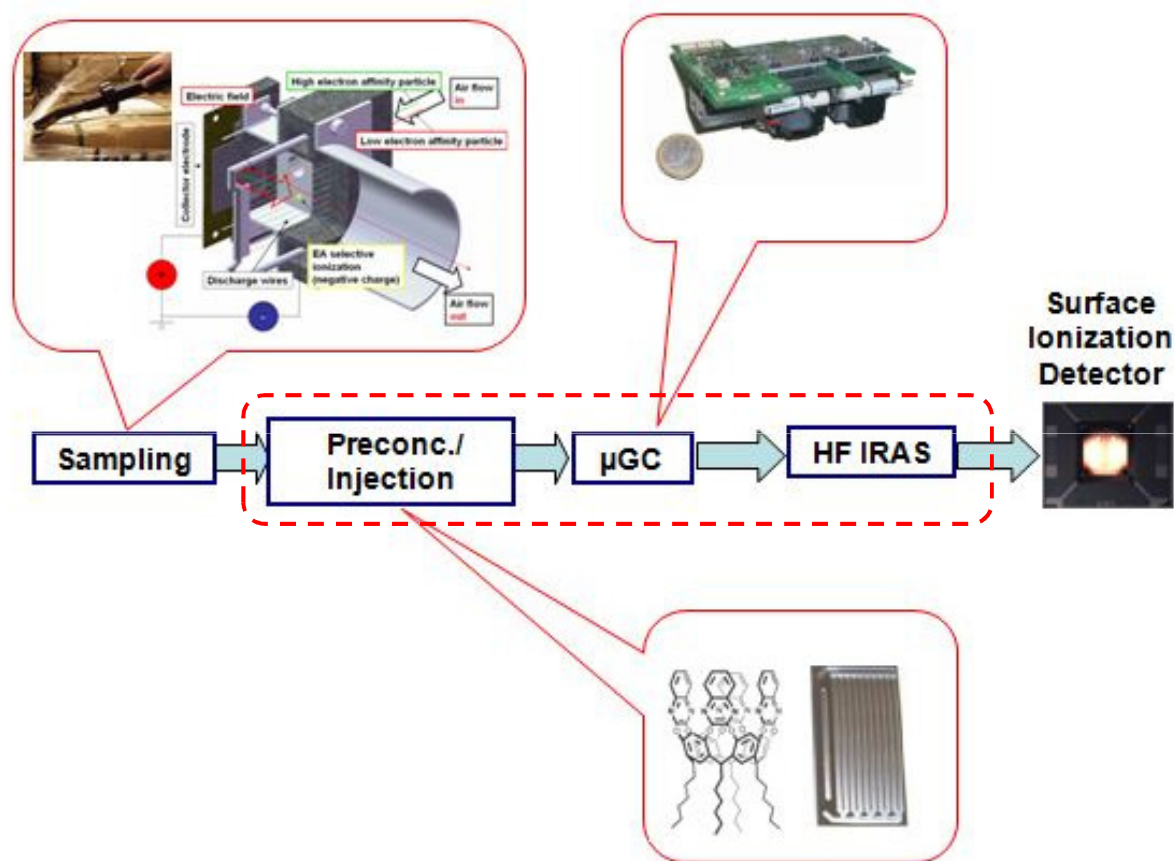
Problems arise over 150° C because of the acrylate coating



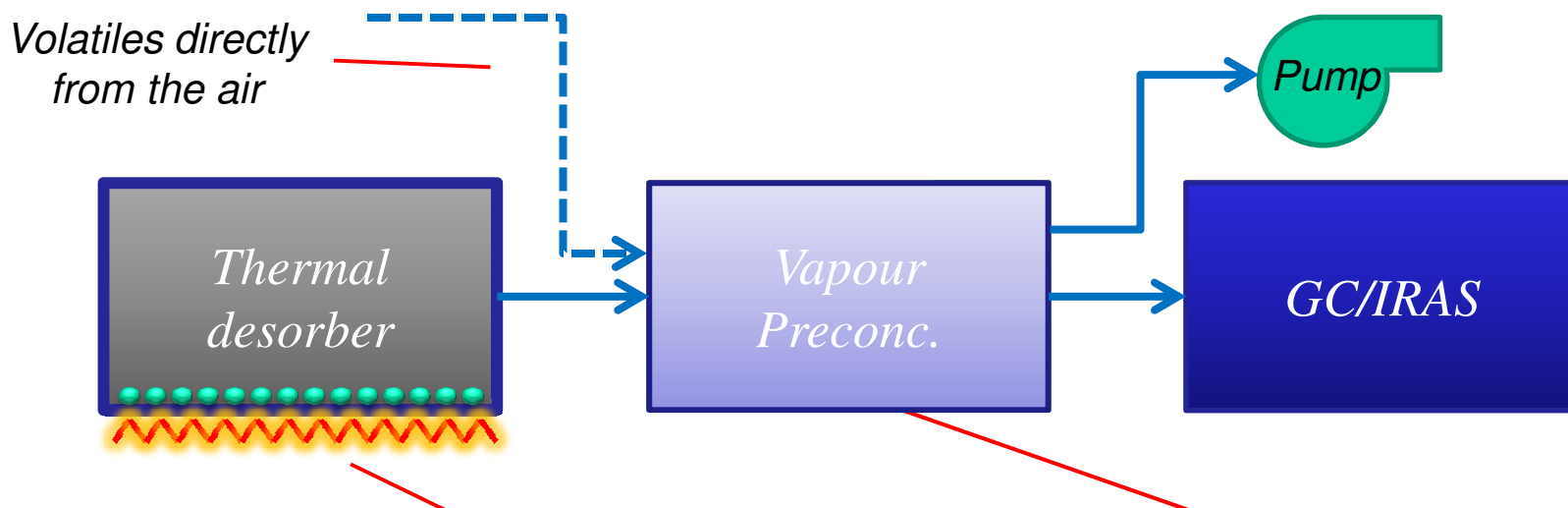
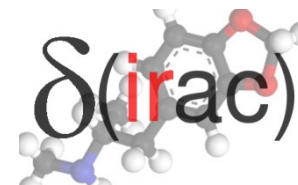
Complex system for the detection and id. of:

- ATSs and precursors;
- Designer drugs
(recognizes analogy with illicit drugs in the 'black list')
- (possibly) Natural drugs;
- as vapours, liquids, and solid particles (salts included);
- as traces and bulk.

The system should have the size of a hand luggage.



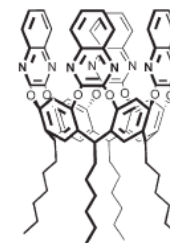
Use of a pre-concentrator in DIRAC



can load:

- Volatile solids (particles), and liquids
- Non volatile **NH⁺ salts** that, dissolved in a basic solution, can be converted into a volatile free amine

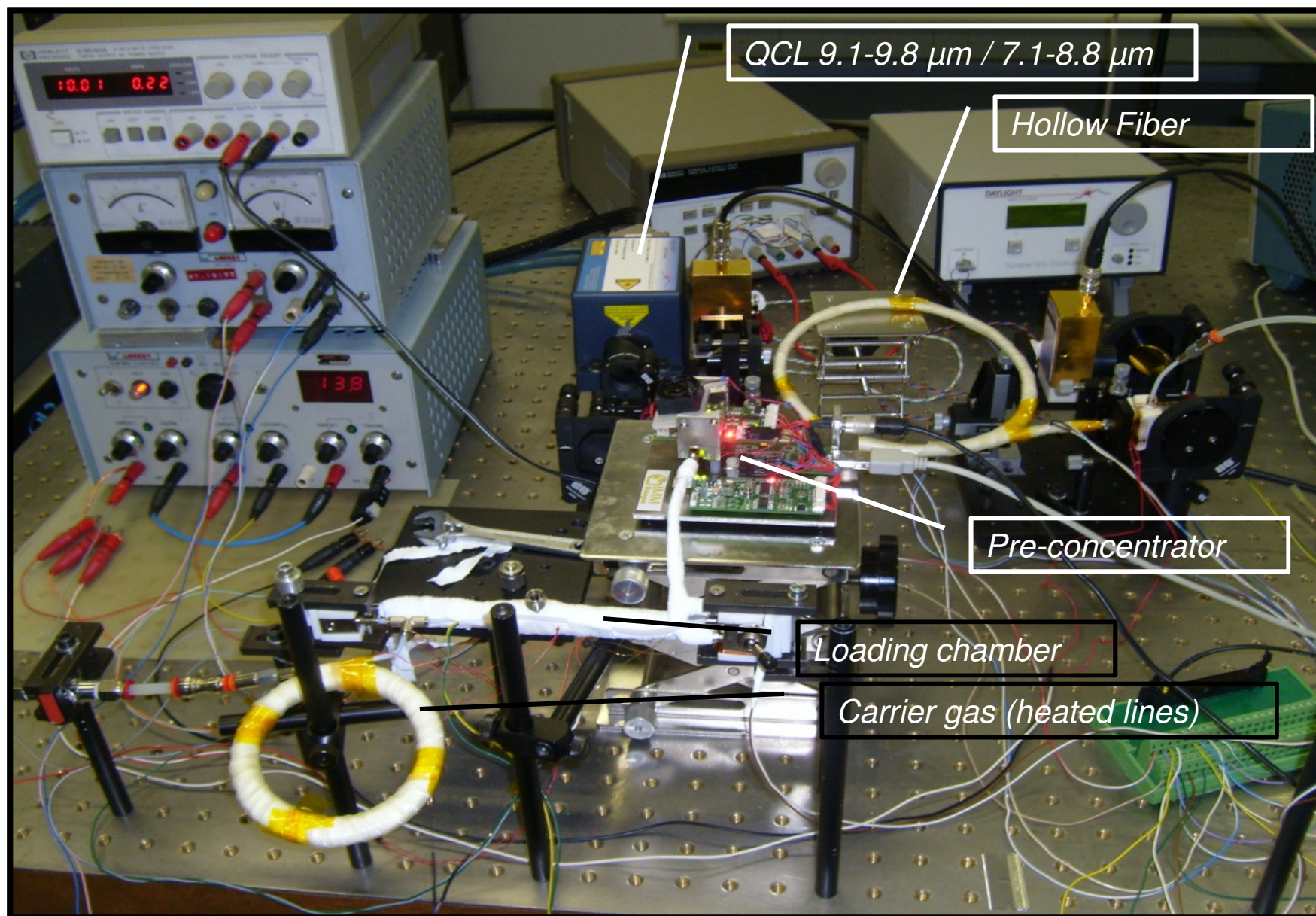
Uses Qx cavitands (right) to selectively trap and pre-concentrate vapours of aromatic compounds on a functionalized Si μ -machined column



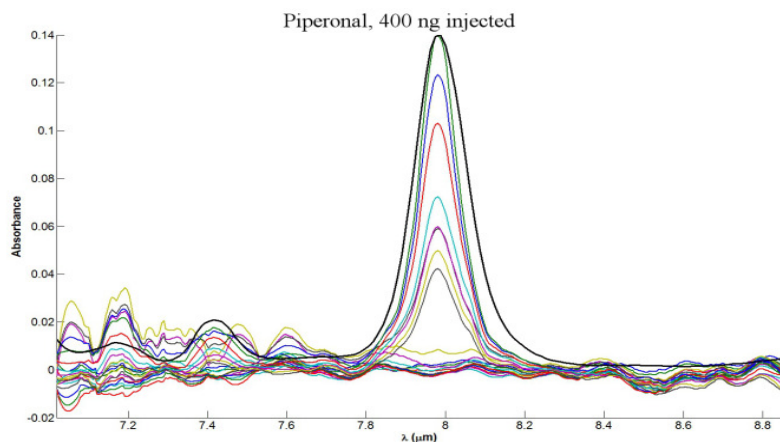
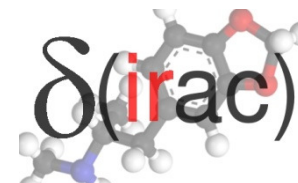
Operations sequence:

- 1) The loading chamber is heated up and vapours are sucked into the pre-concentrator at low temp. (**charging step**);
- 2) The pre-concentrator is heated up with a fast ramp to deliver the trapped vapours to GC/IRAS in a small volume (**release step**)

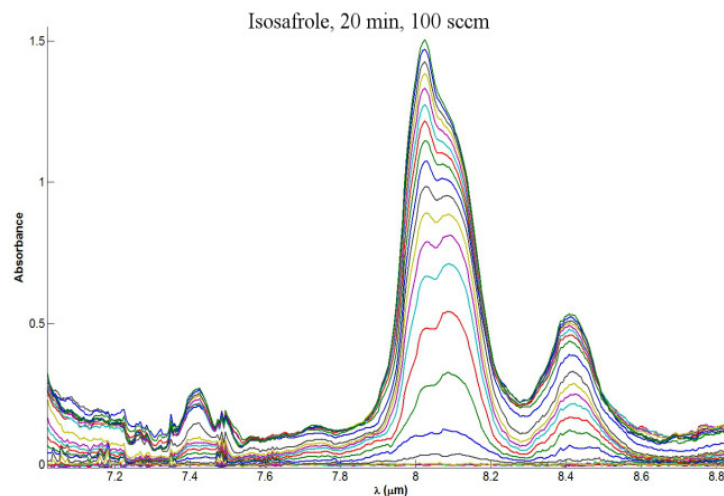
Pre-concentrator + HF-IRAS: set up



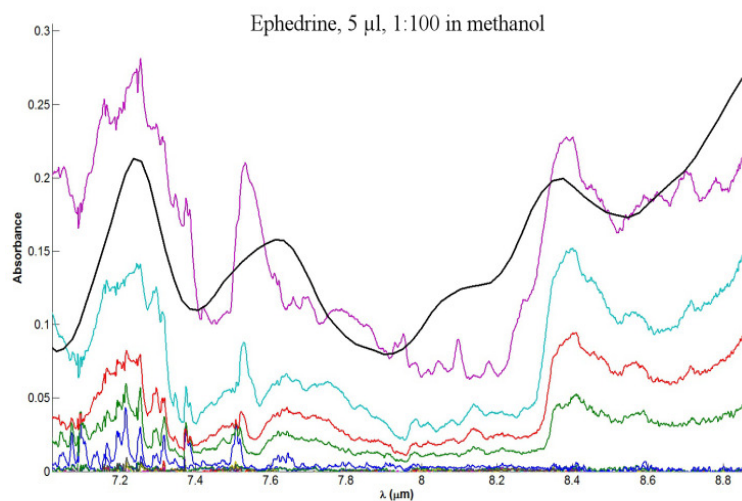
Pre-concentrator + HF-IRAS: results



Piperonal, vaporized from solid grains

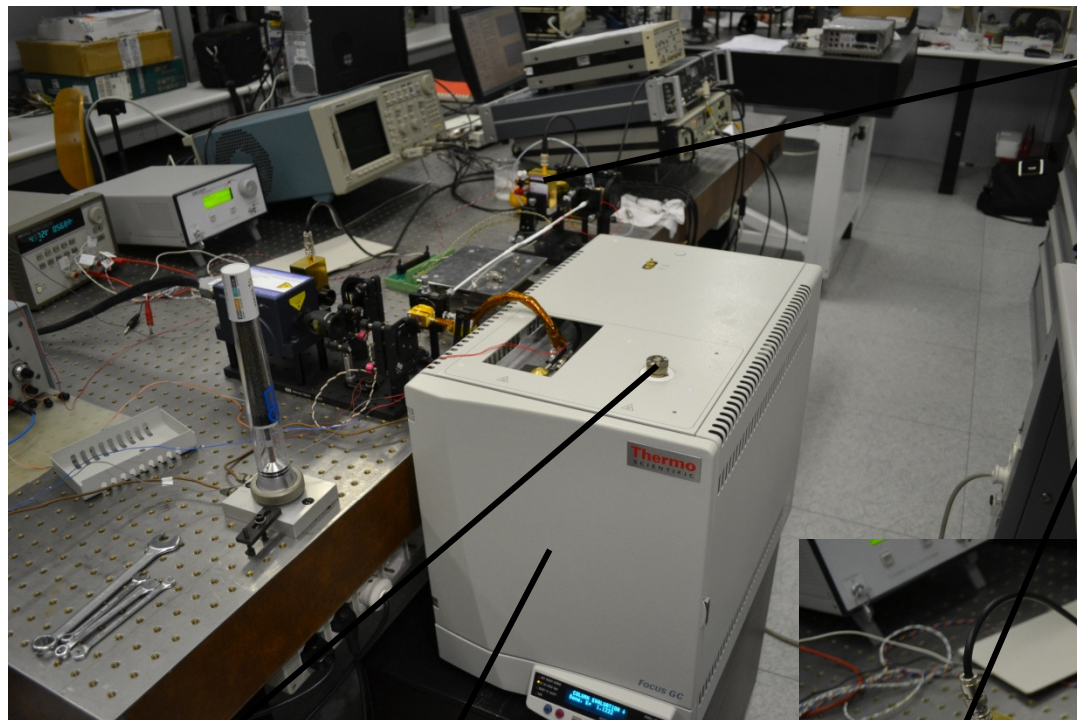
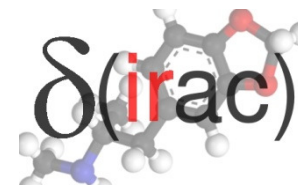


Isosafrole, sampled at room T from the headspace of a vessel containing a few μl



Ephedrine, vaporized in a solution 1:100 of methanol. No evidence of the presence of solvent in the spectrum.

GC + HF-IRAS: set up



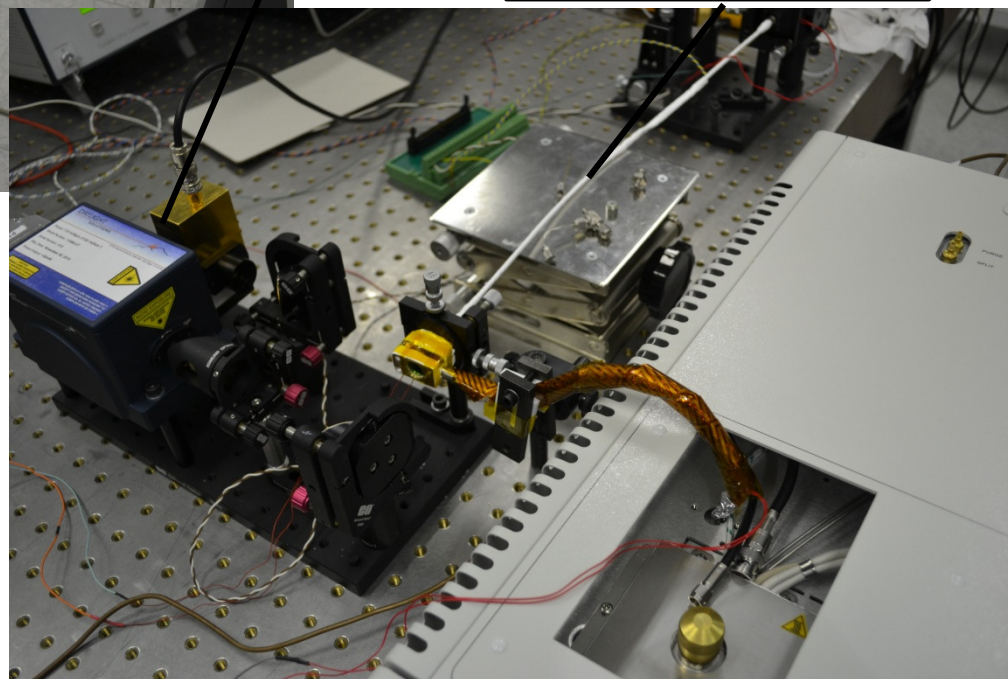
Signal detector
(CMT)

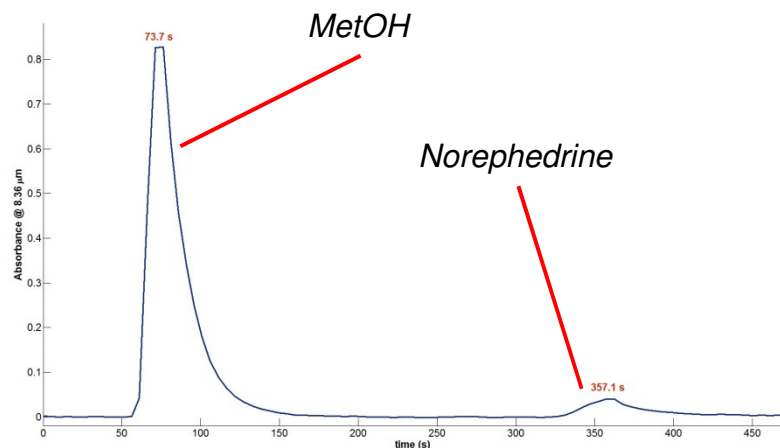
Laser source and
ref. detector

HF phi = 0.5 mm l =
500 mm, with
heating elements

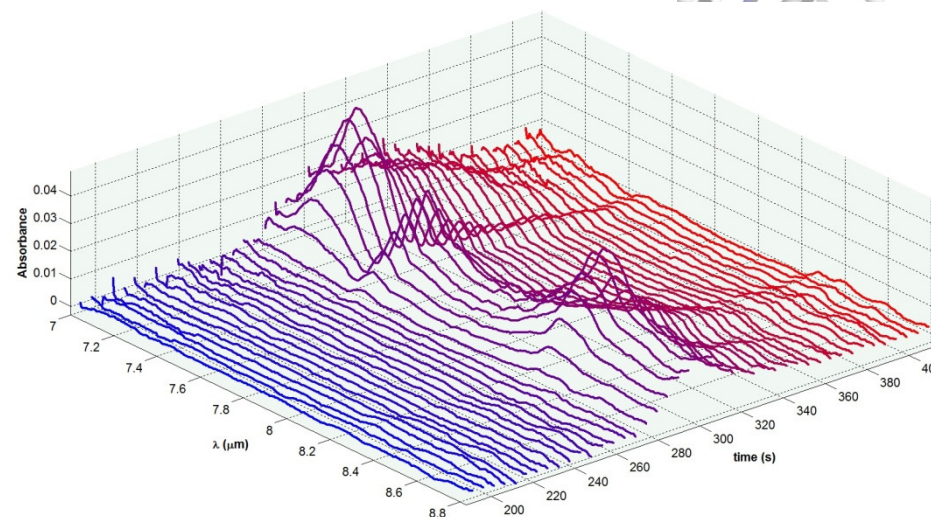
Injection port

Commercial GC; will be
replaced by μ GC on micro-
machined silicon

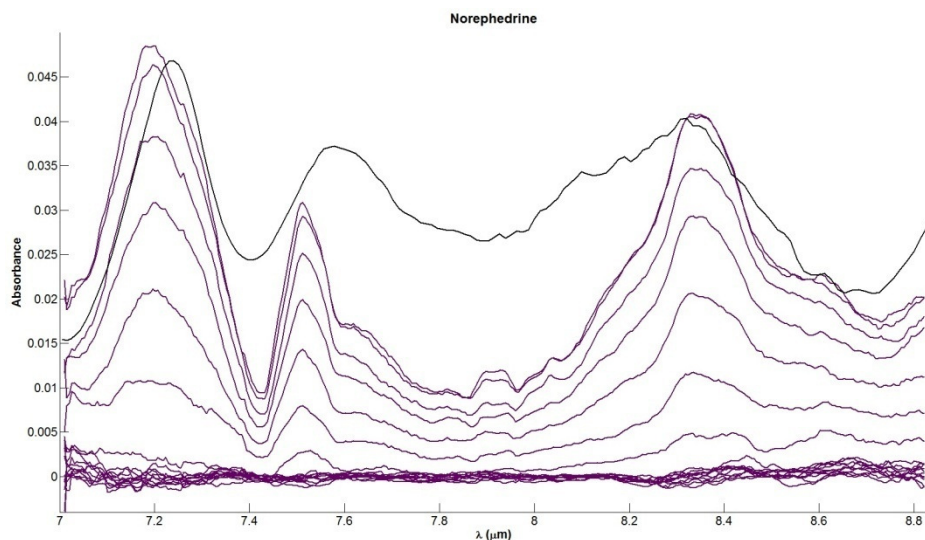




Chromatogram: norephedrine 100g/l in MetOH;
5 μ g injected



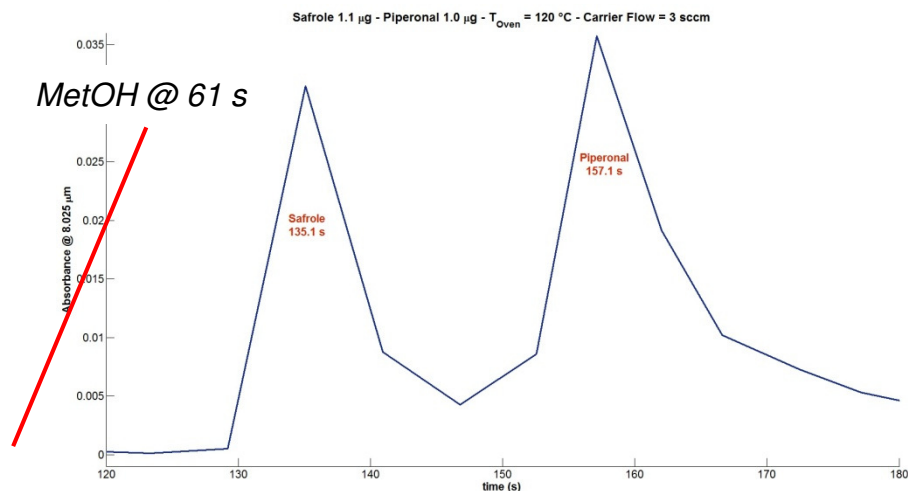
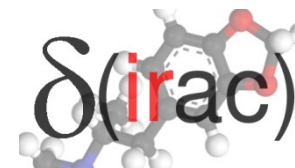
Time dependent Absorption Spectrum



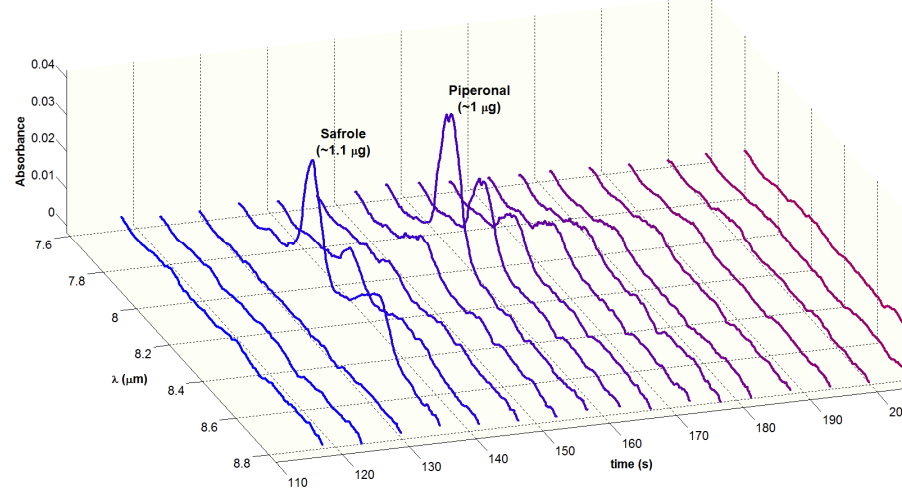
Absorption Spectra around 350 s; Reference Absorption
Spectrum of Norephedrine in black (NIST)

Boiling Point of Norephedrine = 288 °C
Analysis of low volatile chemicals is feasible

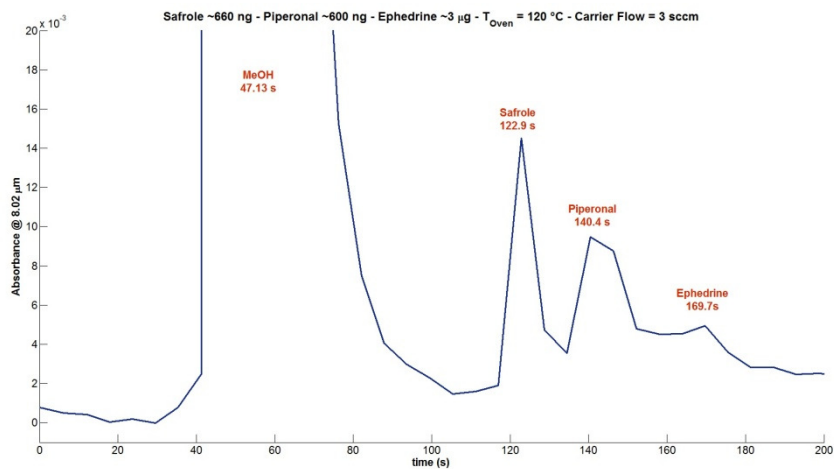
GC + HF-IRAS: precursors mixed in MetOH



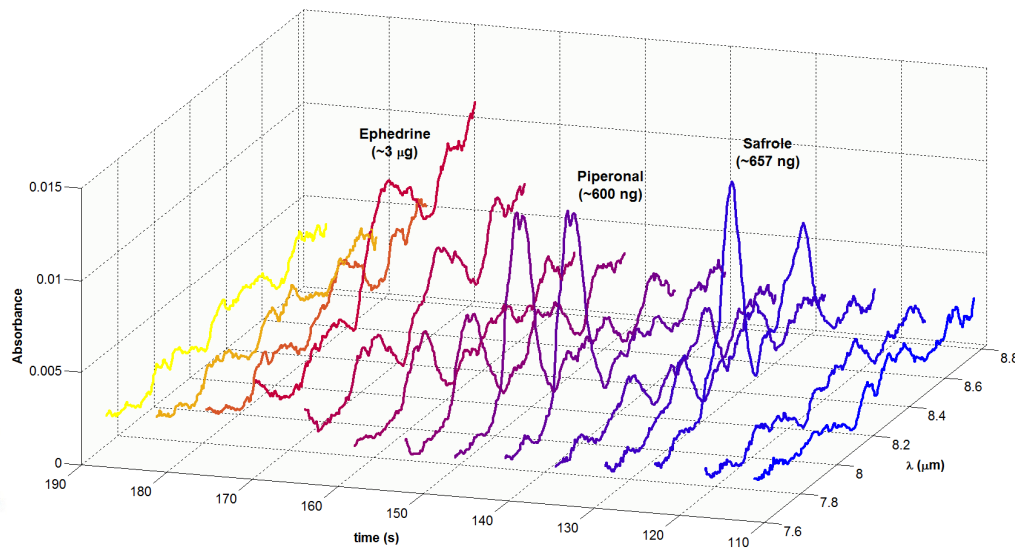
Chromatogram: Safrole and Piperonal in MetOH



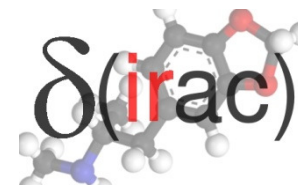
Time dependent Absorption Spectra



Chromatogram: Safrole, Piperonal and Ephedrine in MetOH



Next Steps



- *Test illicit drugs (MA, ECSTASY);*
- *Test precursors and drugs in the presence of excipients and adulterants typical of street samples (lactose, caffeine, paracetamol...);*
- *Optimize process parameters (T ramps, carrier flows..) and determine performance;*
- *Review and finalize design parameters of the Column and the Hollow Fiber;*
- *Fabricate and test μGC .*