Gas-Chromatographic Analysis of Organic Compounds Formed in Avalanches Around Wires

K.Kurvinen, J.Ojala
Helsinki Institute of Physics
00014 University of Helsinki, Finland
T.Särme,
VTT Chemical Technology, Espoo, Finland

Aim

- to analyse stable organic compounds formed in electron avalanches in a proportional counter filled with Ar/ethylene gas mixture.

- previous surveys:

  J.Wise et al., IEEE Trans. Nucl. Sci. NS-35 (2) (1990) 470.

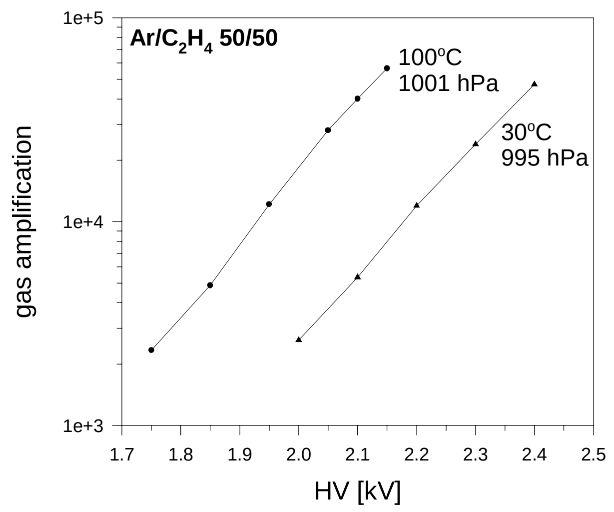
  J.Kadyk, Nucl.Instr. and Meth. A 300 (1991) 436

Content

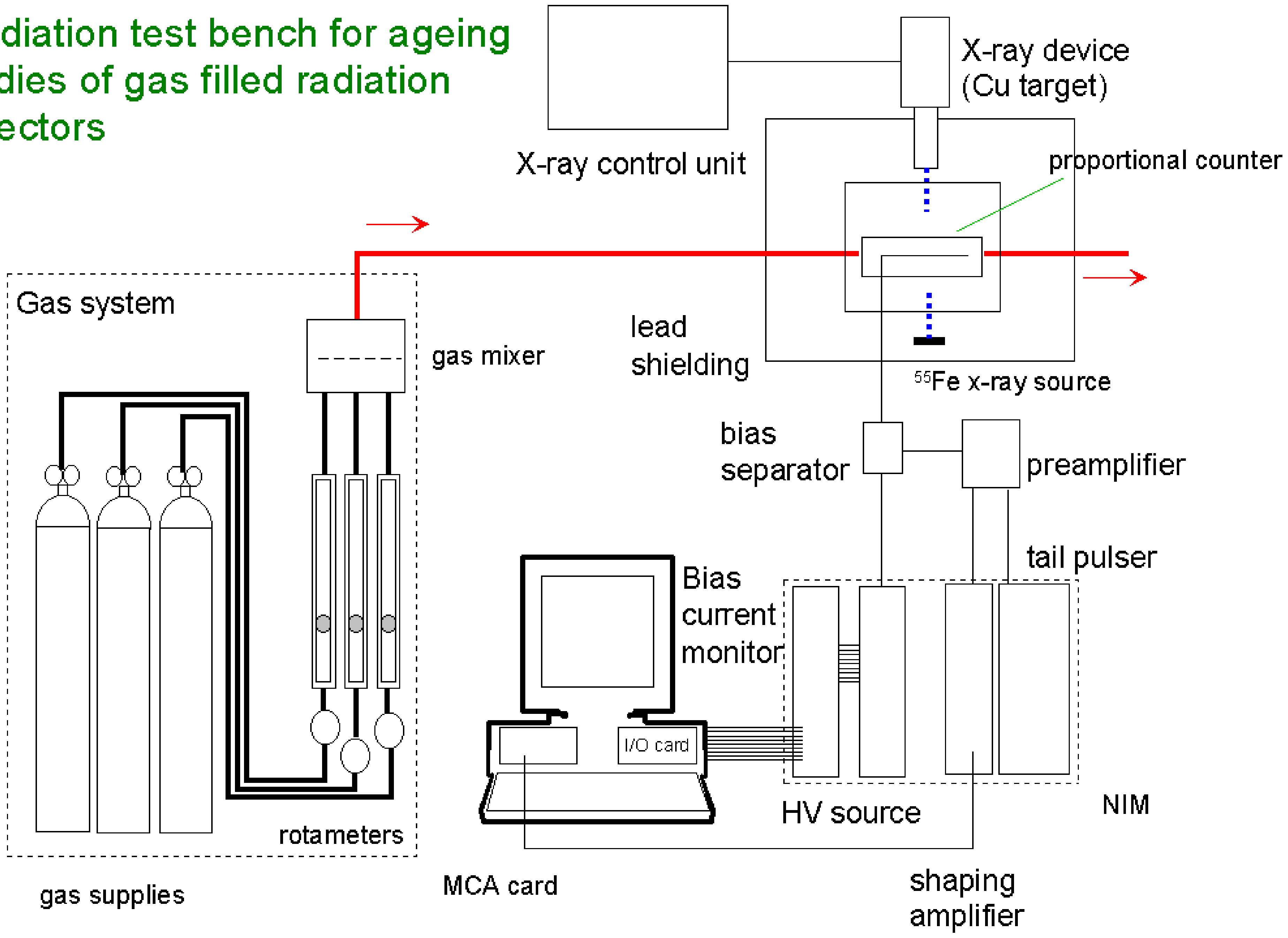
- Description on the gas analysis system and its operation and limitations.
- Analysis of compounds.
- Production of compounds with different irradiation rate.

Detector

- single wire proportional counter
  - gold plated Cu-Be wire (25 μm)
  - other parts: stainless steel, PTFE
  - aluminised Mylar window (clued by Epotek epoxy)
- gas: Ar/C_2H_4 50/50
- temperature: 50 - 70 °C
- gas amplification ~2 x 10^4
- irradiation by X-ray tube (Cu target
- anode current 10 - 500 nA

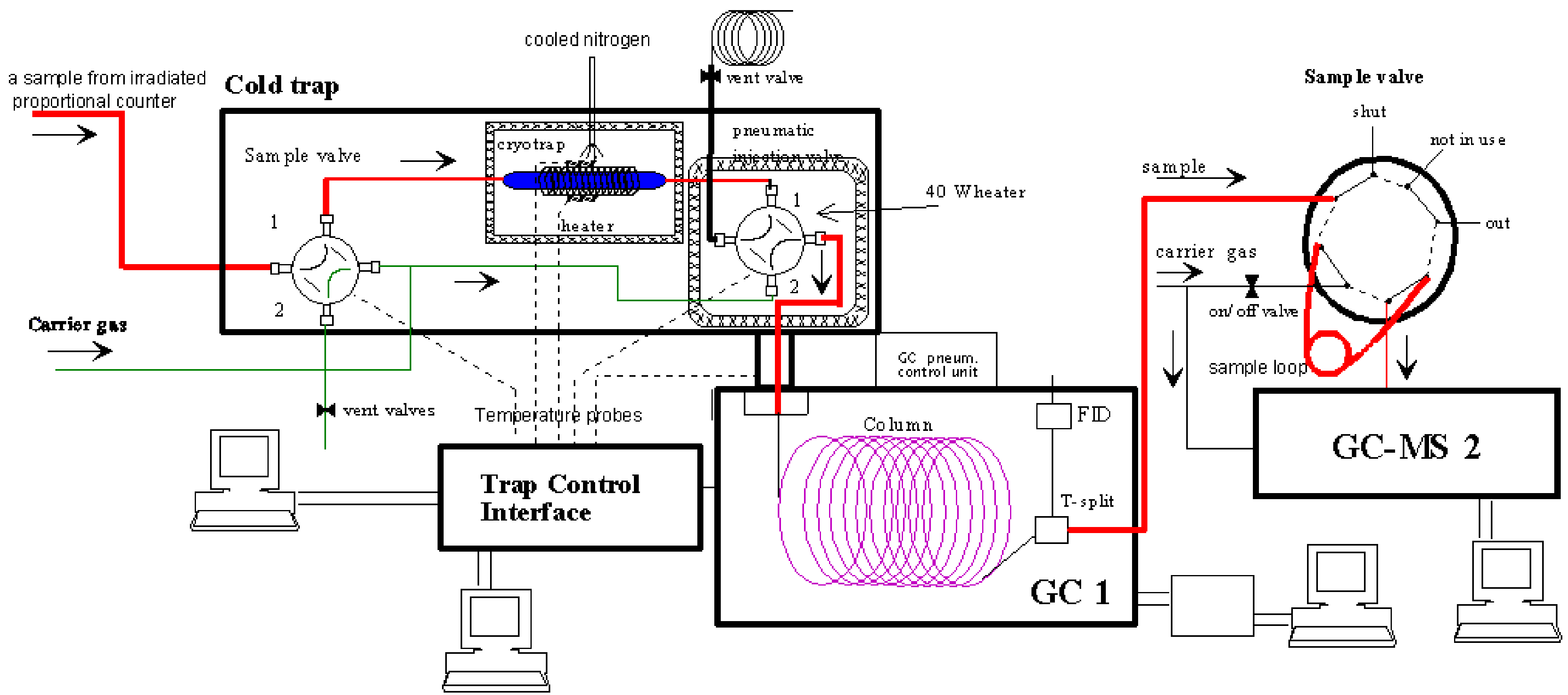


Irradiation test bench for ageing studies of gas filled radiation detectors



Gas analysis system for ageing studies of gaseous radiation detectors

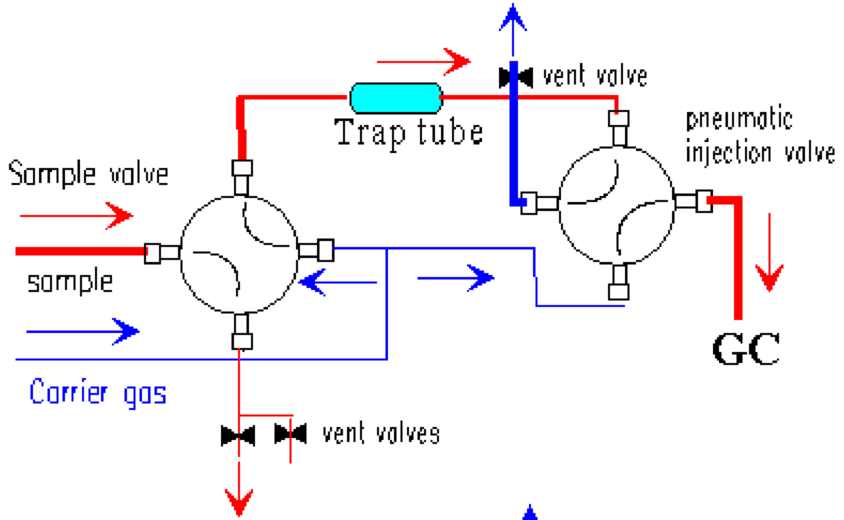
Kari Kurvinen
Jukka Ojala
Timo Särme



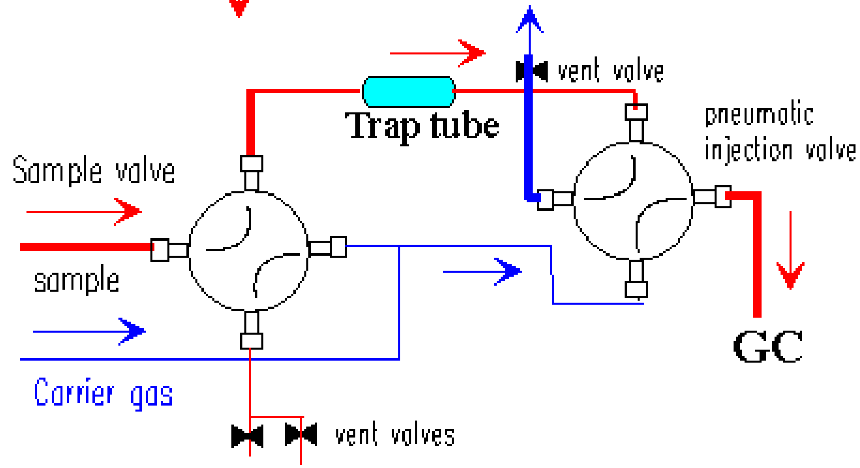
- sample concentration by cryotrap (for heavier compounds than quenching gas)
- tandem gas chromatograph: first GC for quantitative analysis, second GC-MS for identification
- for analysis of organic compounds formed in electron avalanches or outgassed from detector materials

Trapping sequences

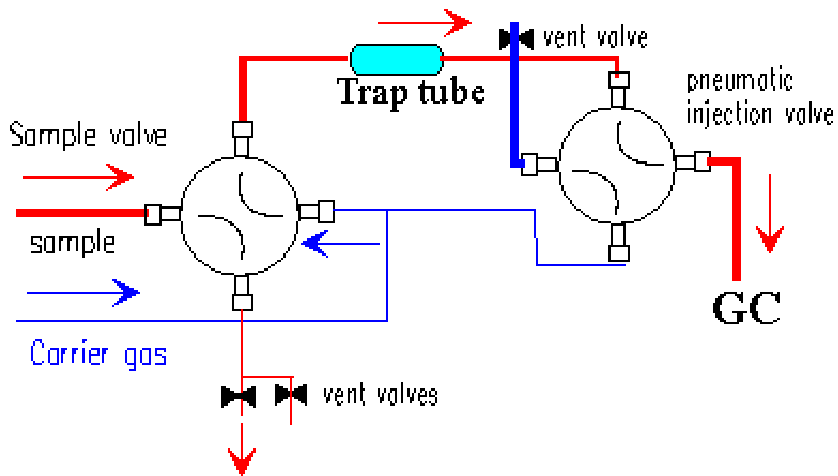
I Phase Purge / Cooling



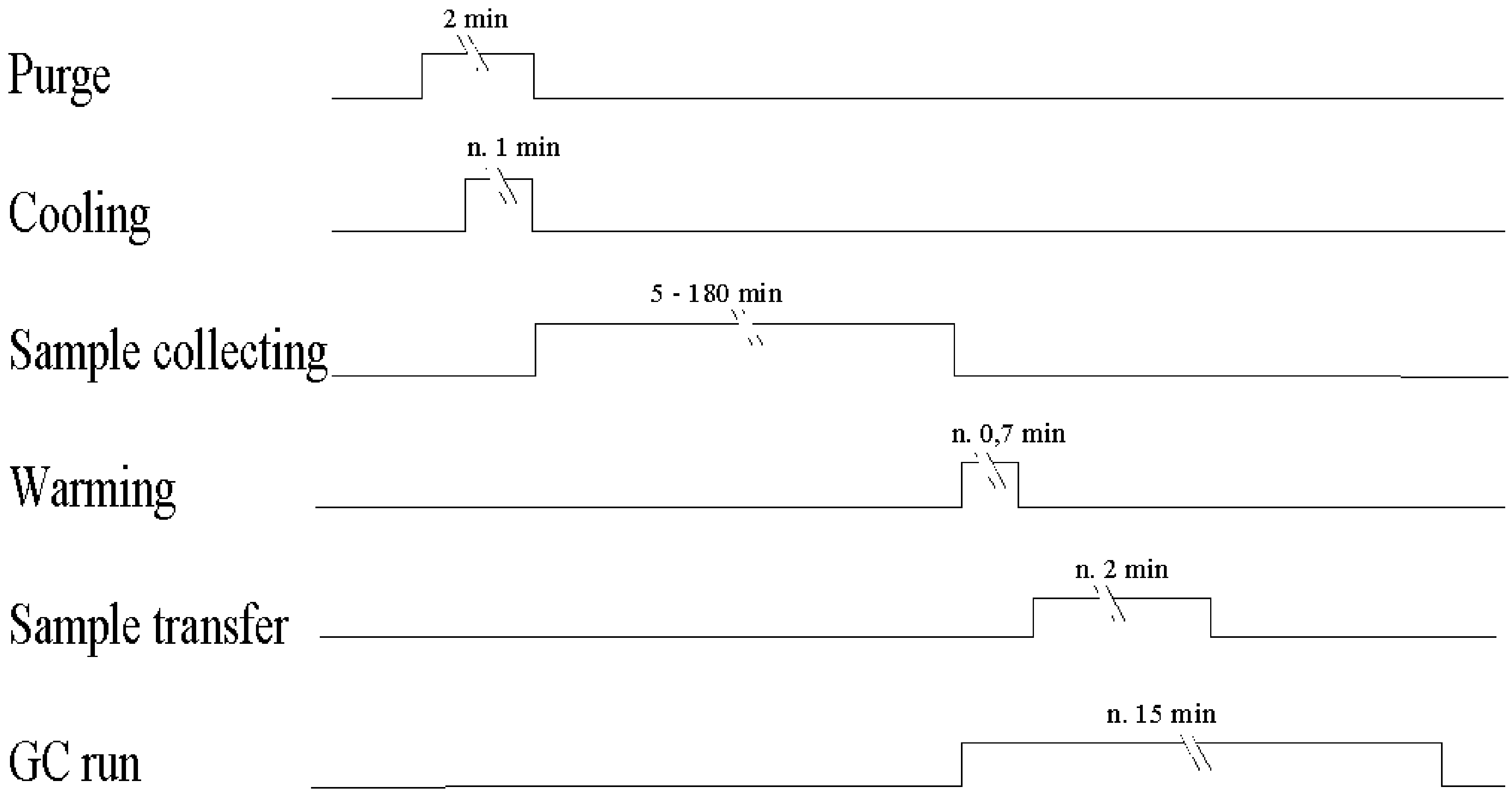
II Phase Sample Trapping



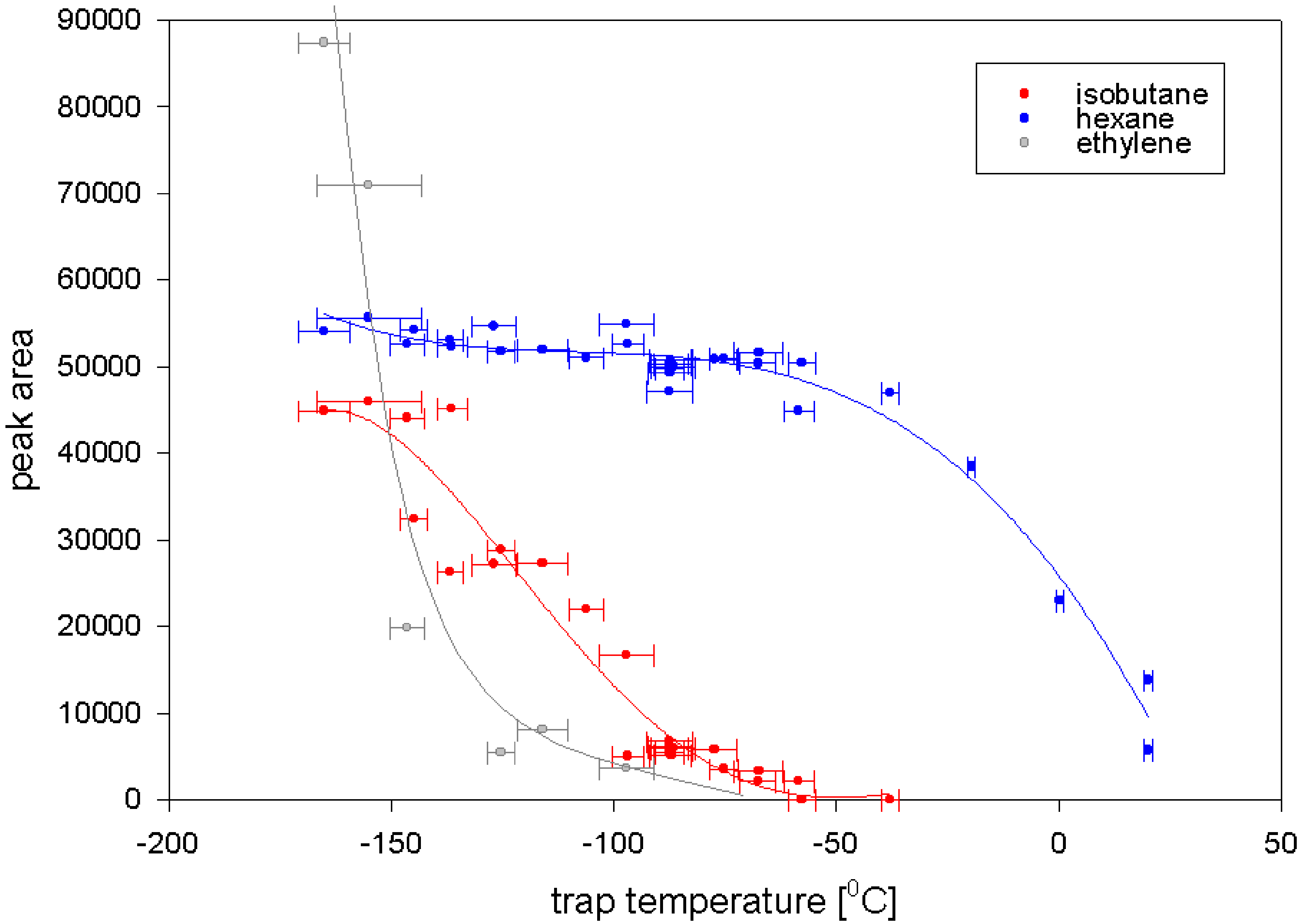
III Phase Sample Injection to GC



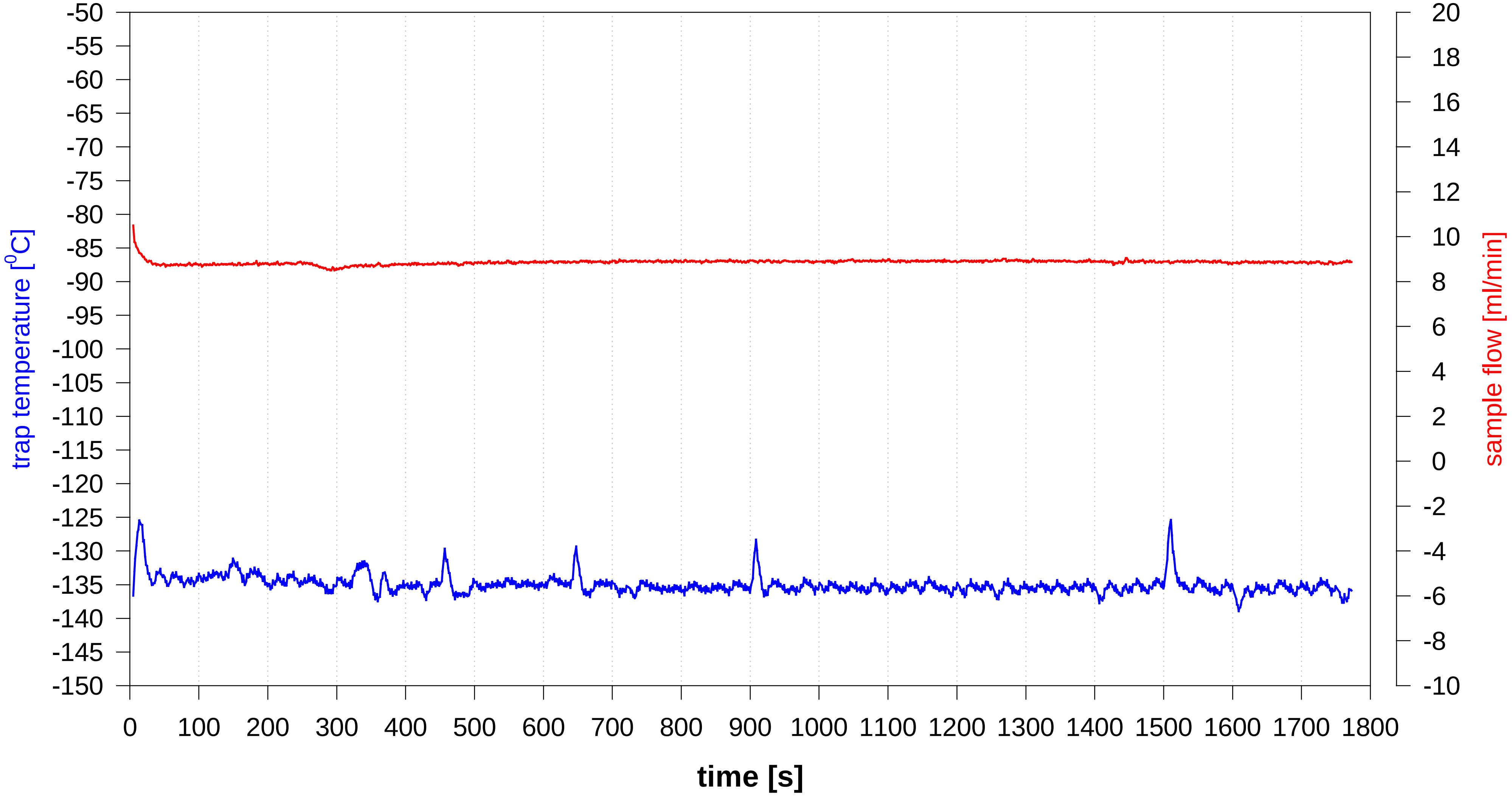
Timing diagram of cryotrapping



Trapping efficiency vs. trapping temperature



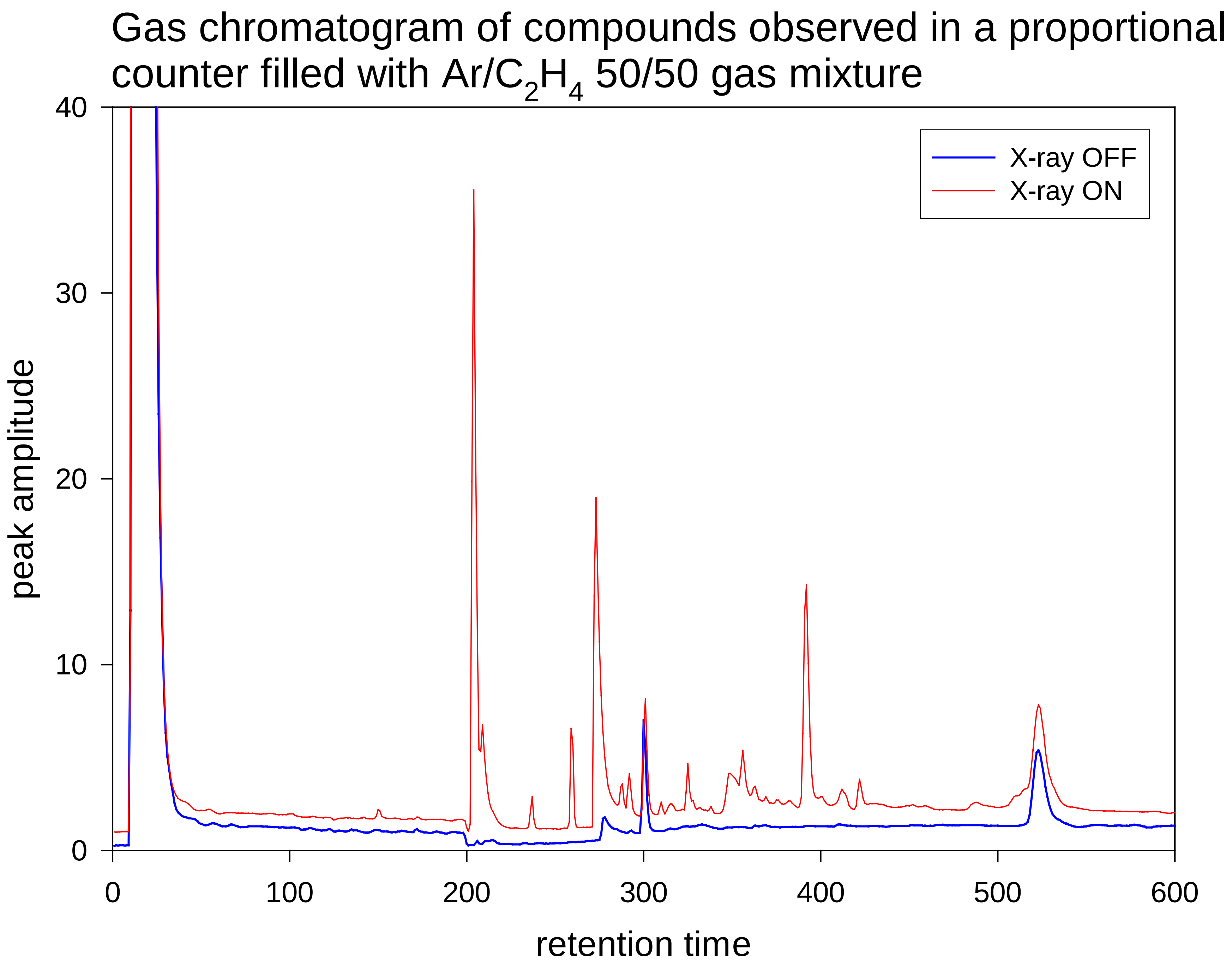
Trap temperature and sample flow during trapping



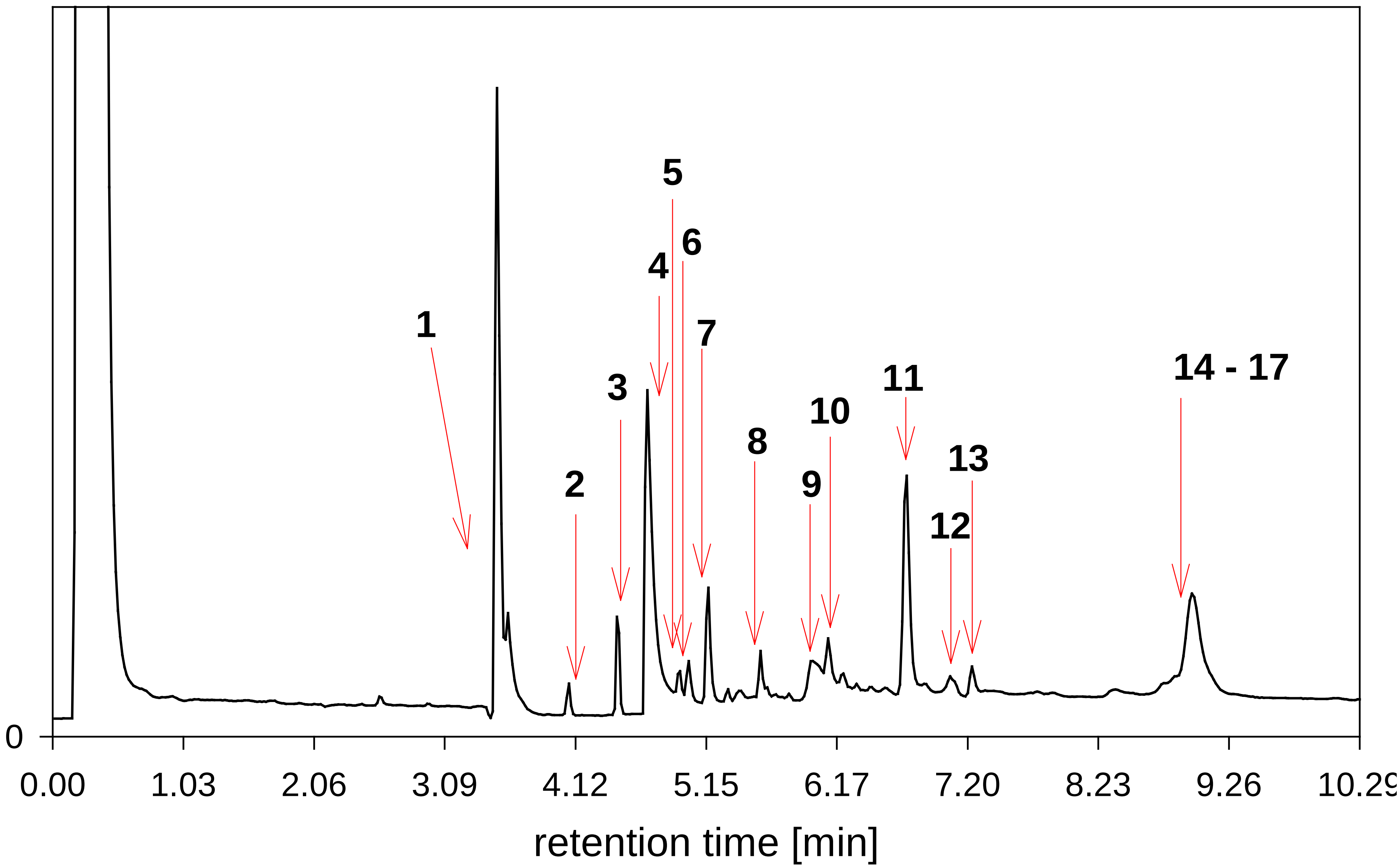
LTRAP156

Limitations of system

- detects just <u>stable organic</u> compounds heavier than quenching gas.
- detector in elevated temperature.
- sensitivity ~ 1 ng (depends in trapping time and compounds).
- complicated and time consuming operation.


Gas chromatogram of compounds observed in a proportional counter filled with Ar/C2H4 50/50 gas mixture
X-ray OFF
X-ray ON
peak amplitude
retention time
0
10
20
30
40
0
100
200
300
400
500
600

Some identified compounds created in electron avalanches in proportional mode with Ar/C_2H_4 50/50 gas mixture



Avalanche compounds identified

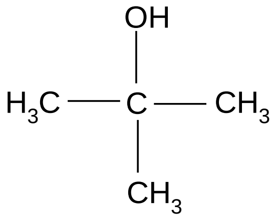
PEAK	COMPOUND	SOURCE	REMARK
1	Asetaldehyde	Electron aval.	Polymerising improbable.
2	1,3-butadiyne	Electron aval.	Explosively polymerising.*
3	Ethanol	Electron aval.	Polymerising improbable.
4	1,3-pentadiene	Electron aval.	Able to polymerise.
5	2-methyl-2-propanol	Electron aval.	Polymerising improbable.
6	Methoxy-asetaldehyde	Electron aval.	Polymerising improbable.
7	2-ethoxy-2-methylpropane	From system.	Polymerising improbable.
8	2-methyl-1,3-dioxolane	Electron aval.	Polymerising improbable.
9	2-methoxy-ethanol	Electron aval.	Polymerising improbable.
10	1,3-hexadien-5-yne	Electron aval.	Able to polymerise.
11	3-methyl-1,3-pentadiene	Electron aval.	Able to polymerise.
12	4-methyl-1,4-hexadiene	Electron aval.	Able to polymerise.
13	2,4-heptadiene	Electron aval.	Able to polymerise.
14	Tetracloroethylene	From gas bottle.	Contaminant in ethylene bottle.
15	1-ethenyl-4-ethylbenzene	Electron aval.	Able to polymerising.
16	2,3-dihydro-1-methylindene	Electron aval.	Polymerising improbable.
17	4-ethylbenzaldehyde	Electron aval.	Polymerising improbable.

* "Potentially very explosive, it may be handled and transferred by low temperature distillation. It should be stored at -25 ^{0}C to prevent decomposition and formation of explosive polymers." *(Armitage, J.B. et al.,* J.Chem.Soc., 1951, 44)

Ethylene + water

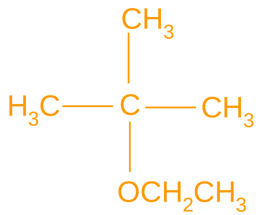
$2\ CH_2{=}CH_2 + 2H_2O \longrightarrow 2\ CH_3CH_2OH$ ethanol (peak3)

$2\ CH_2{=}CH_2 + H_2O \rightarrow \ \rightarrow$



2-methyl-2-propanol (peak 5)

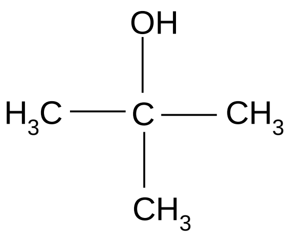
$3\ CH_2{=}CH_2 + H_2O \rightarrow \ \rightarrow$



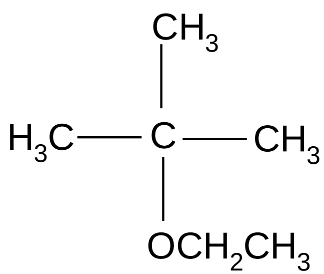
2-ethoxy-2-methyl-propane (peak 7)

Impurity from syst

(or by secondary reactions:



$+ \ CH_3CH_2OH \rightleftarrows \ \rightleftarrows H_2O +$



)

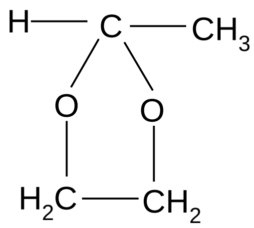
Ethylene + Oxygen

$2\ CH_2{=}CH_2 + O_2 \longrightarrow 2\ CH_3CHO$ (asetaldehyde) (peak 1)

$2\ CH_2{=}CH_2 + O_2 \rightarrow \rightarrow H_3C\text{-}O\text{-}CH_2\text{-}CHO + :CH_2$ (peak 6)

methoxy-asetaldehyde (+carbene radical react further)

$2\ CH_2{=}CH_2 + O_2 \rightarrow \rightarrow$



2-methyl-1,3-dioxolane (peak 8)

$2\ CH_2{=}CH_2 + O_2 \rightarrow \rightarrow CH_3\text{-}O\text{-}CH_2\text{-}CH_2\text{-}OH + C$ (peak 9)

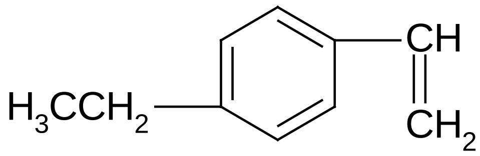
2-methoxy-ethanol

(or by secondary reactions:

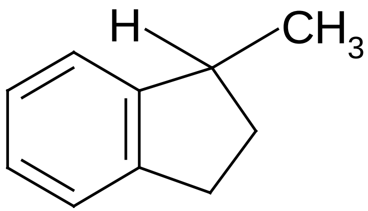
$CH_4 + \frac{1}{2}O_2 + CH_3\text{-}CH_2\text{-}OH \rightarrow \rightarrow CH_3\text{-}O\text{-}CH_2\text{-}CH_2\text{-}OH + H_2$

$CH_3OH + CH_3\text{-}CH_2\text{-}OH \rightarrow \rightarrow CH_3\text{-}O\text{-}CH_2\text{-}CH_2\text{-}OH + H_2$)

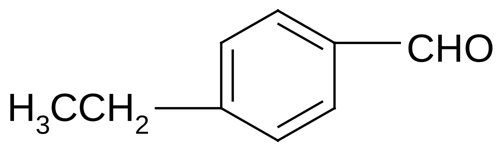
Aromatic hydrocarbons

$5\ CH_2{=}CH_2 \rightarrow \rightarrow 4\ H_2 +$ 

1-ethenyl-4-ethyl-benzene (peak 15)

$5\ CH_2{=}CH_2 \rightarrow \rightarrow 4\ H_2 +$ 

2,3-dihydro-1-methylindene (peak 16)

$10\ CH_2{=}CH_2 + O_2 \rightarrow \rightarrow 2CH_4 + 6H_2 + 2$ 

4-ethyl-benzaldehyde (peak 17)

Aliphatic hydrocarbons

(Initial chaining?)

$2\ CH_2 = CH_2 \rightarrow \rightarrow HC \equiv C\text{-}C \equiv CH + 3\ H_2$ 1,3-butadiyne (Peak 2)

$3\ CH_2 = CH_2 \rightarrow \rightarrow CH_2 = CH\text{-}CH = CH\text{-}CH_3 + CH_4$

1,3-pentadiene (Peak 4)

$3\ CH_2 = CH_2 \rightarrow \rightarrow CH_2 = CH\text{-}CH = CH\text{-}C \equiv CH + 3H_2$

1,3-hexadien-5-yne (Peak 10)

$3\ CH_2 = CH_2 \rightarrow \rightarrow CH_2 = CH\text{-}C(\text{-}CH_3) = CH\text{-}CH_3 + H_2$

3-methyl-1,3-pentadiene (Peak 11)

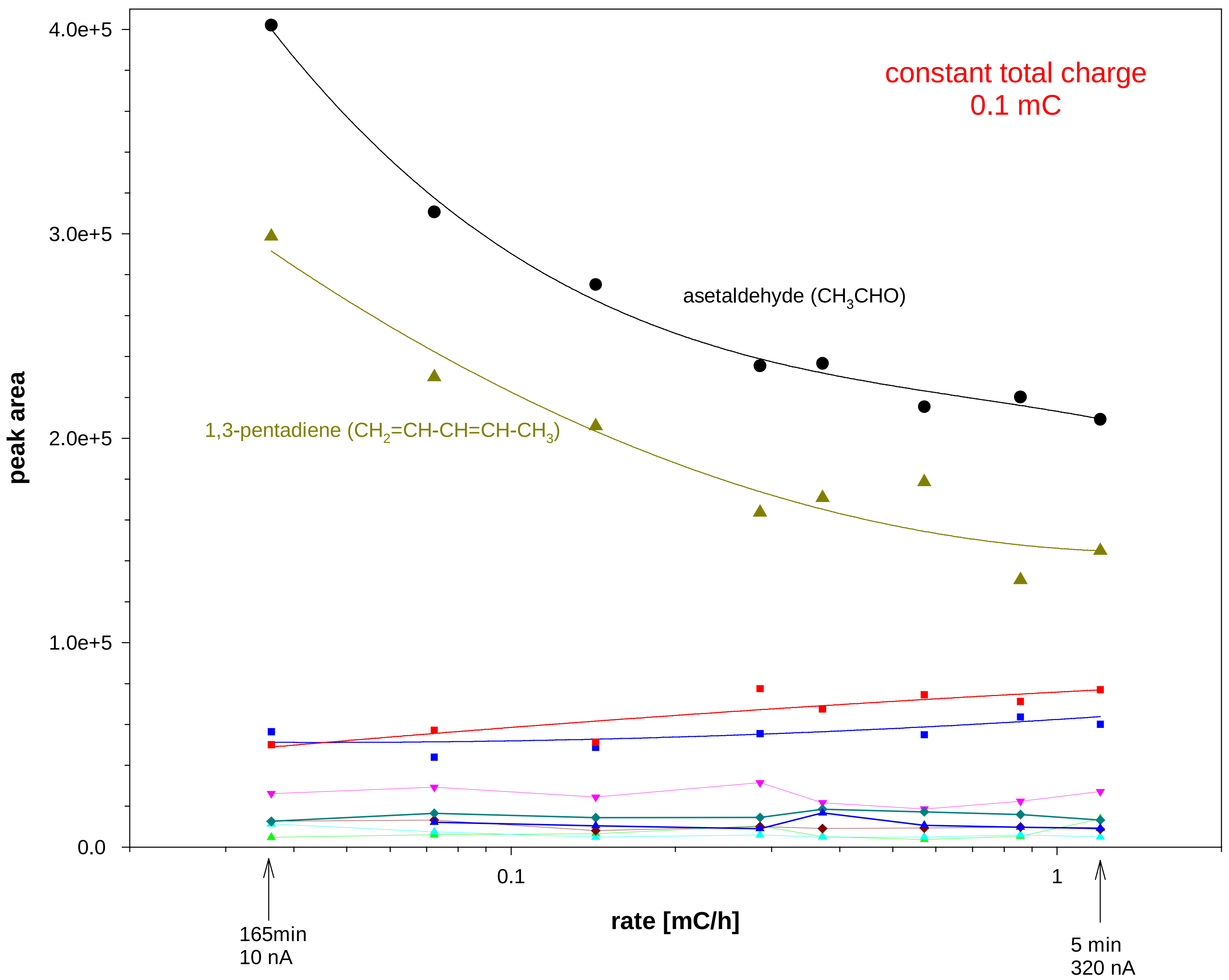
$4\ CH_2 = CH_2 \rightarrow \rightarrow CH_2 = CH\text{-}CH_2\text{-}C(\text{-}CH_3) = CH\text{-}CH_3 + CH_4$

4-methyl-1,4-hexadiene (Peak 12)

$4\ CH_2 = CH_2 \rightarrow \rightarrow CH_3\text{-}CH = CH\text{-}CH = CH\text{-}CH_2\text{-}CH_3 + CH_4$

2,4-heptadiene (Peak 13)

Rate dependence of production of avalanche compounds (Ar/C_2H_4 50/50)



Remark for accelerated aging tests

- in accelerated aging tests irradiation rates are increased by factor of several hundreds.
- molarity should be taken into account for those impurity compounds which are supposed to react with avalanche compounds.

$$A + I \rightarrow C$$

compound from avalanche

additive / impurity

?

compound not from avalanche

$$B + I \rightarrow D$$

Summary

- Some organic compounds formed in electron avalanches in Ar/C_2H_4 50/50 gas mixture analysed and identified.

Future

- effect of O_2
- effect of additives and outgassing components.
- new gas mixtures (P-10)