HELSINKI INSTITUTE OF PHYSICS

RD-51 WG2 meeting 10th Dec. 2008

Ageing tests and analysis of organic compounds released from various detector materials

Kari Kurvinen on behalf of

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based on talks given in NSS 2003 and NSS2004 symposium

(see conf.CDs and IEEE Trans. on Nucl. Sci 51 No.5, 2004)

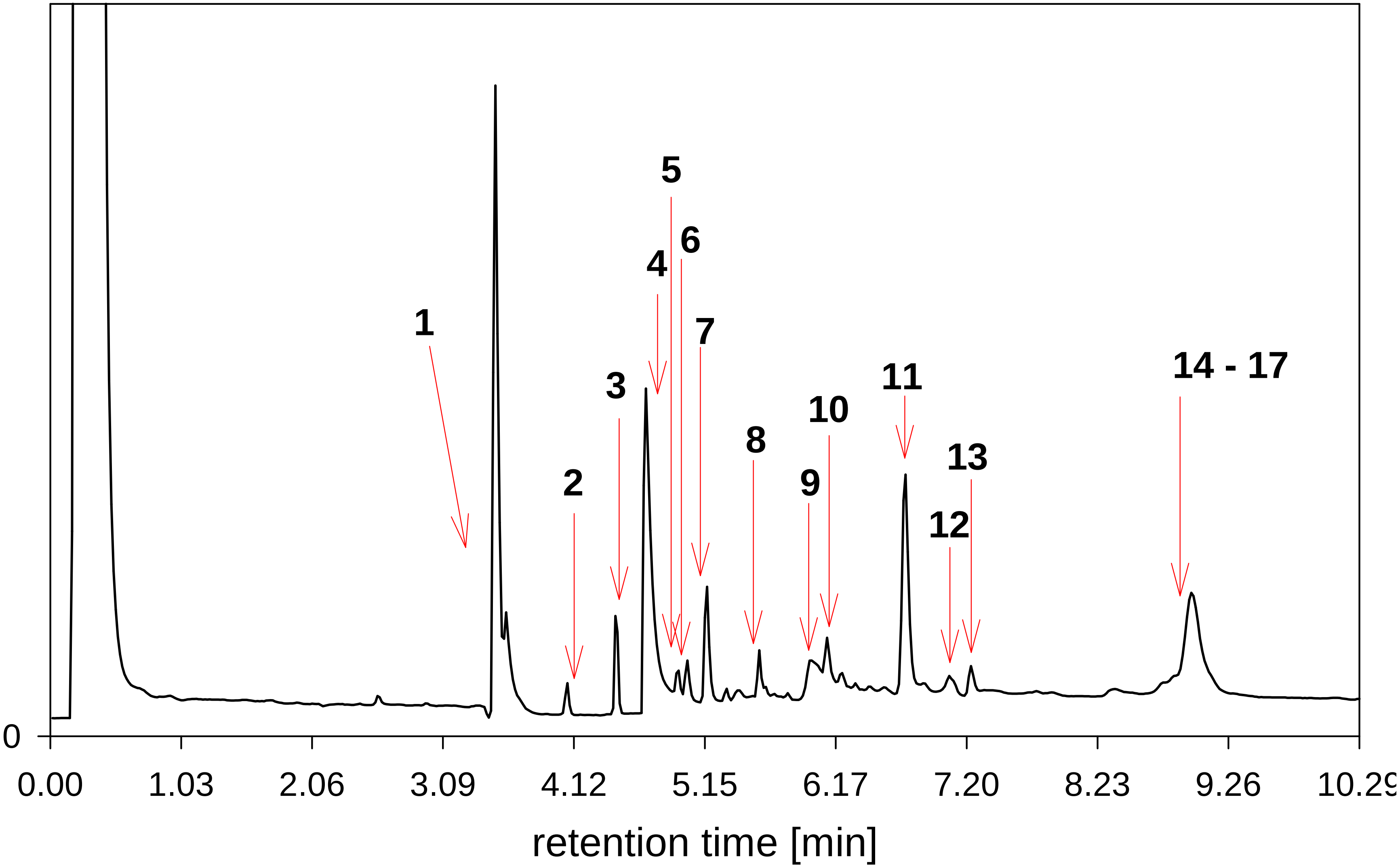
Outline

- on wire chamber chemistry
- classical methods of outgassing and ageing tests
- compound oriented ageing tests
- some materials analysed and **organic** compounds found
- ageing tests with an array of proportional counters

Wire chamber chemistry:

sample concentration by a cold trap

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



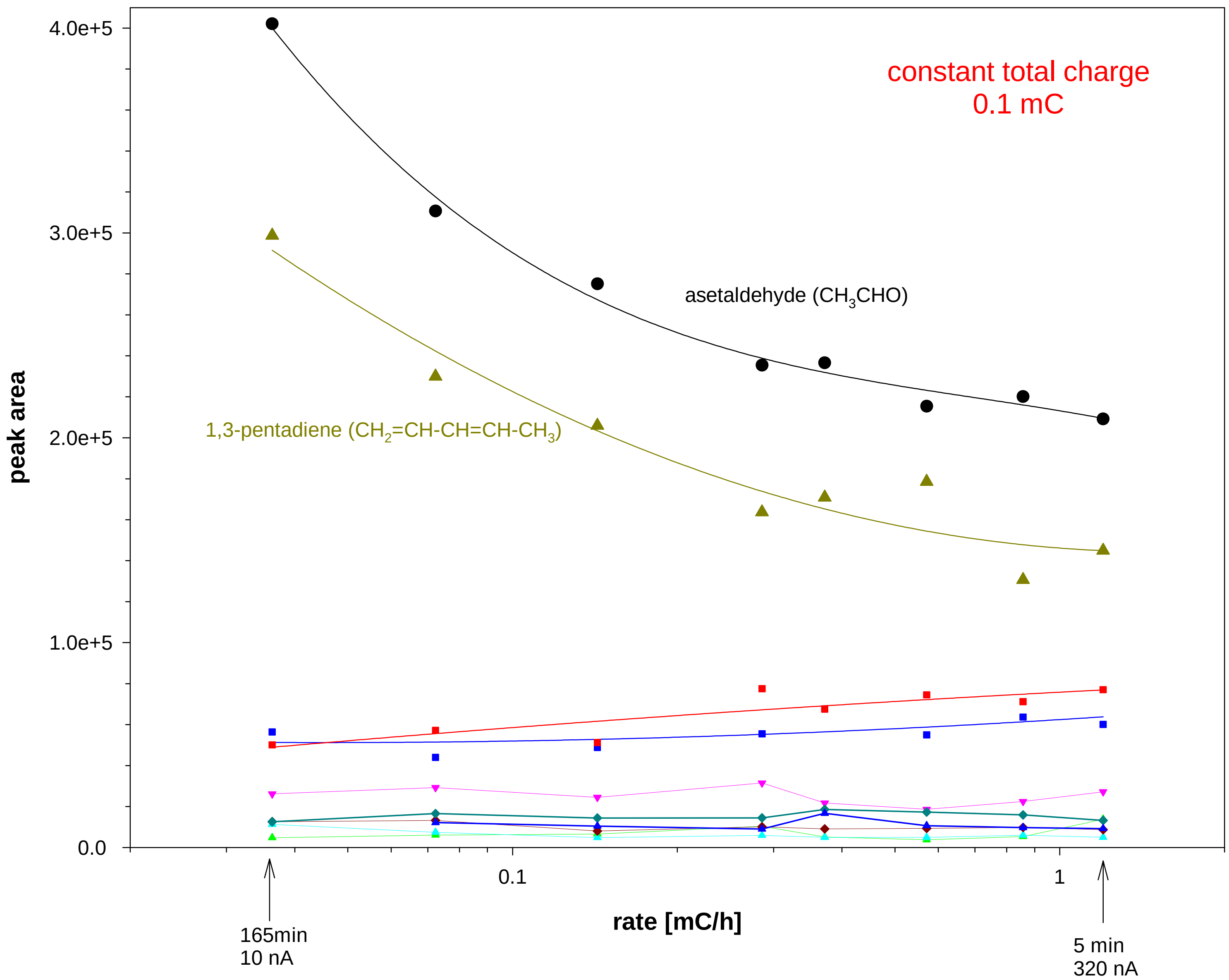
Wire chamber chemistry:

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.

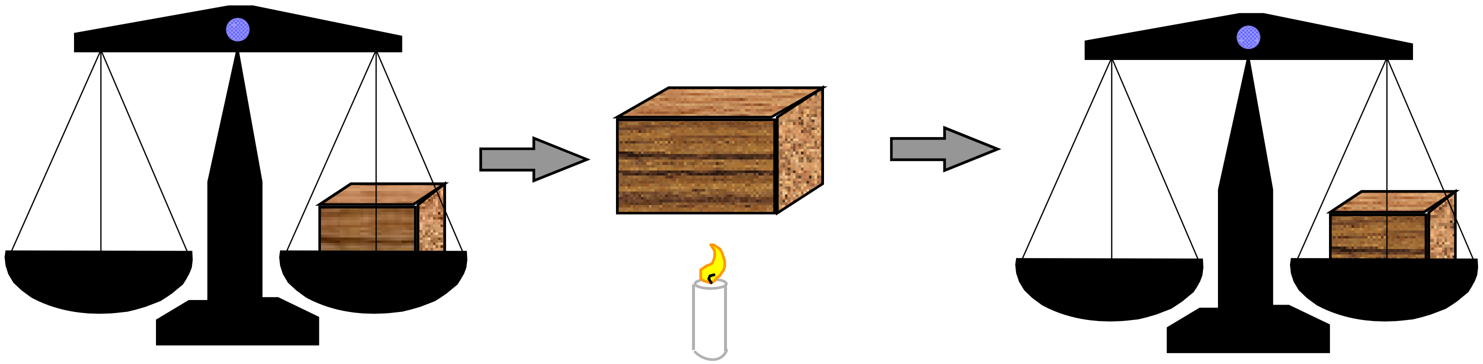
* “Potentially very explosive, it may be handled and transferred by low temperature distillation. It should be stored at -25 ^{0}C to prevent decomposition and formation of explosive polymers.” *(Armitage, J.B. et al.*, J.Chem.Soc., 1951, 44)

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



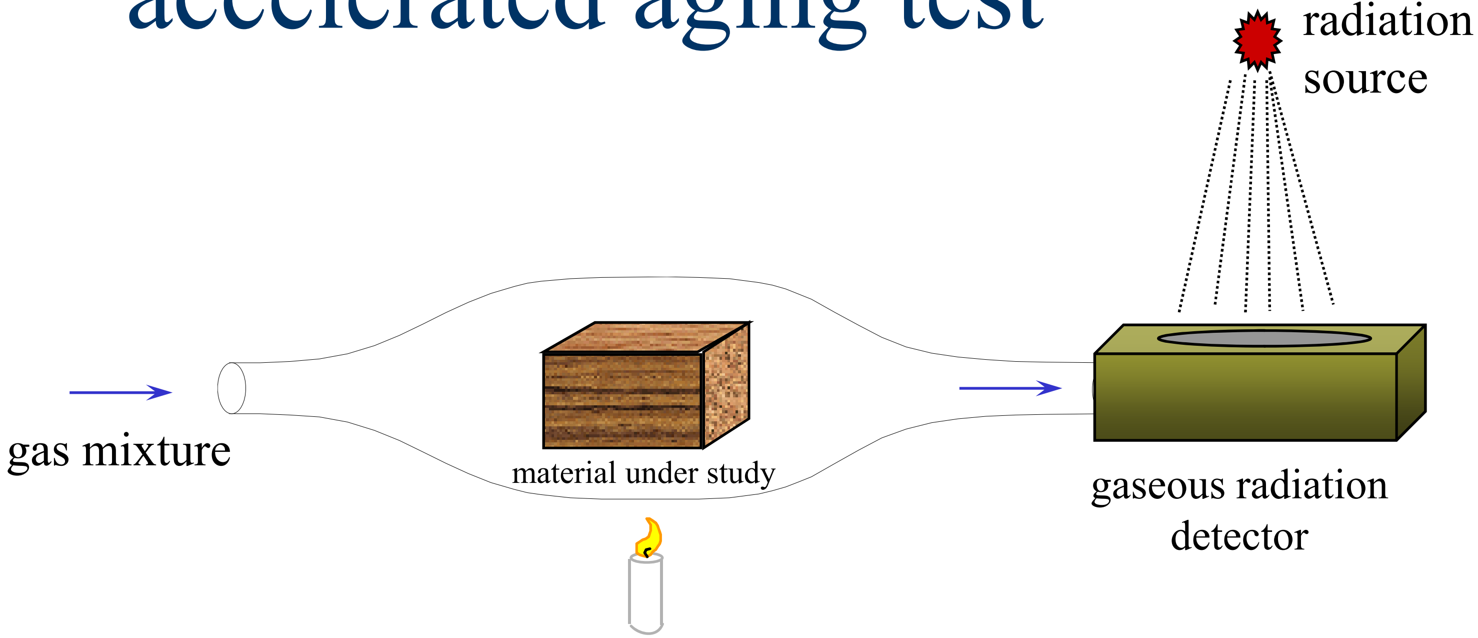
Standard outgassing test

weight loss method



- the only information obtained is the mass loss during the heating.
- no information about the outgassed substances.
- outgassing in the standard room temperature must be extrapolated.

Outgas study with an accelerated aging test



• monitoring current, gas gain, energy resolution, etc.

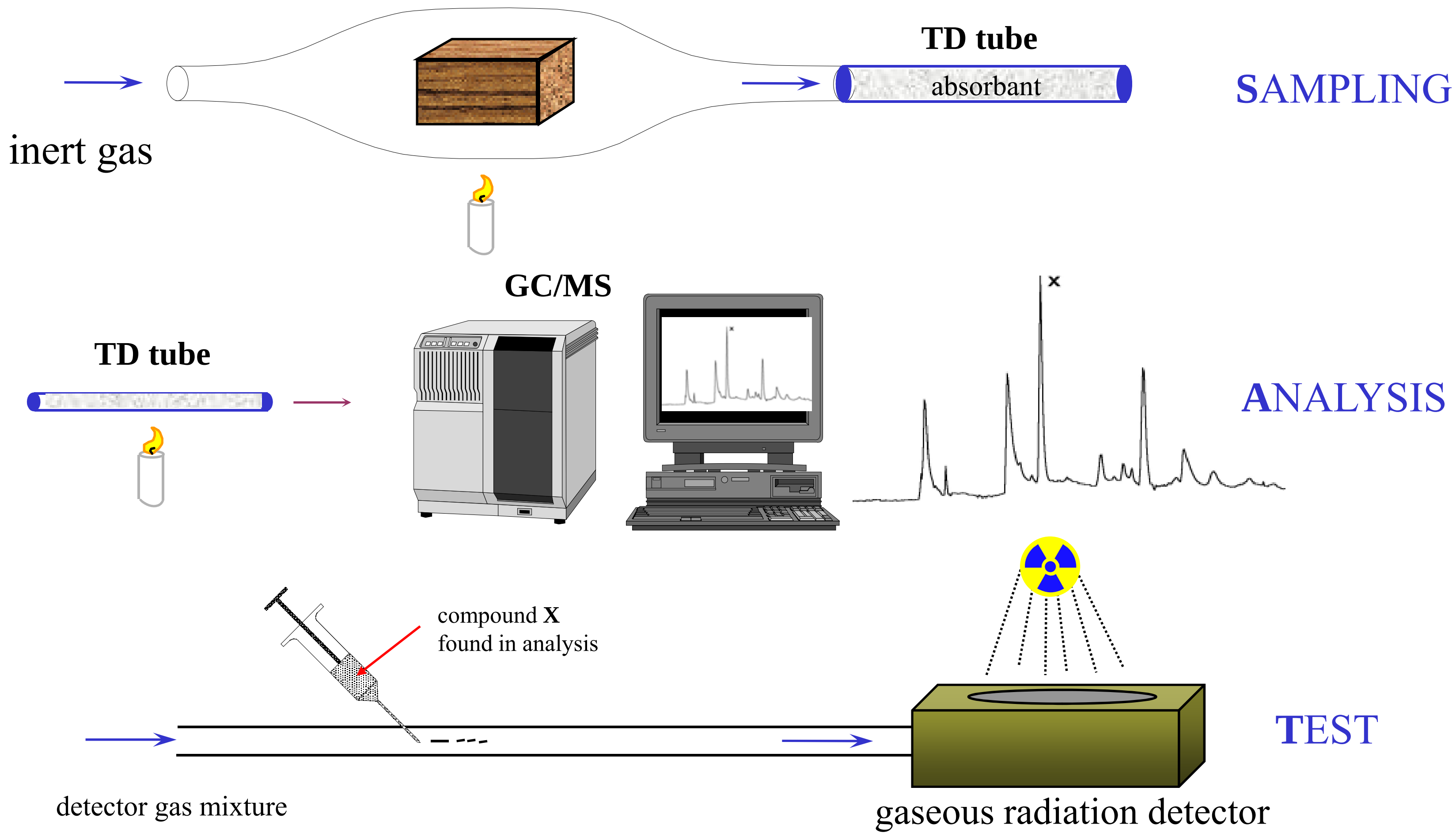
database of harmful ***materials***

Classical accelerated aging test

- benefits
  - gives definite information of combatibility of the material with the detector
- drawbacks
  - laborious and time consuming method
  - for each new material and new manufacturer of old materials tests must be repeated.
  - no information about the aging process itself.

Introduction

Combined outgas analysis and accelerated aging test



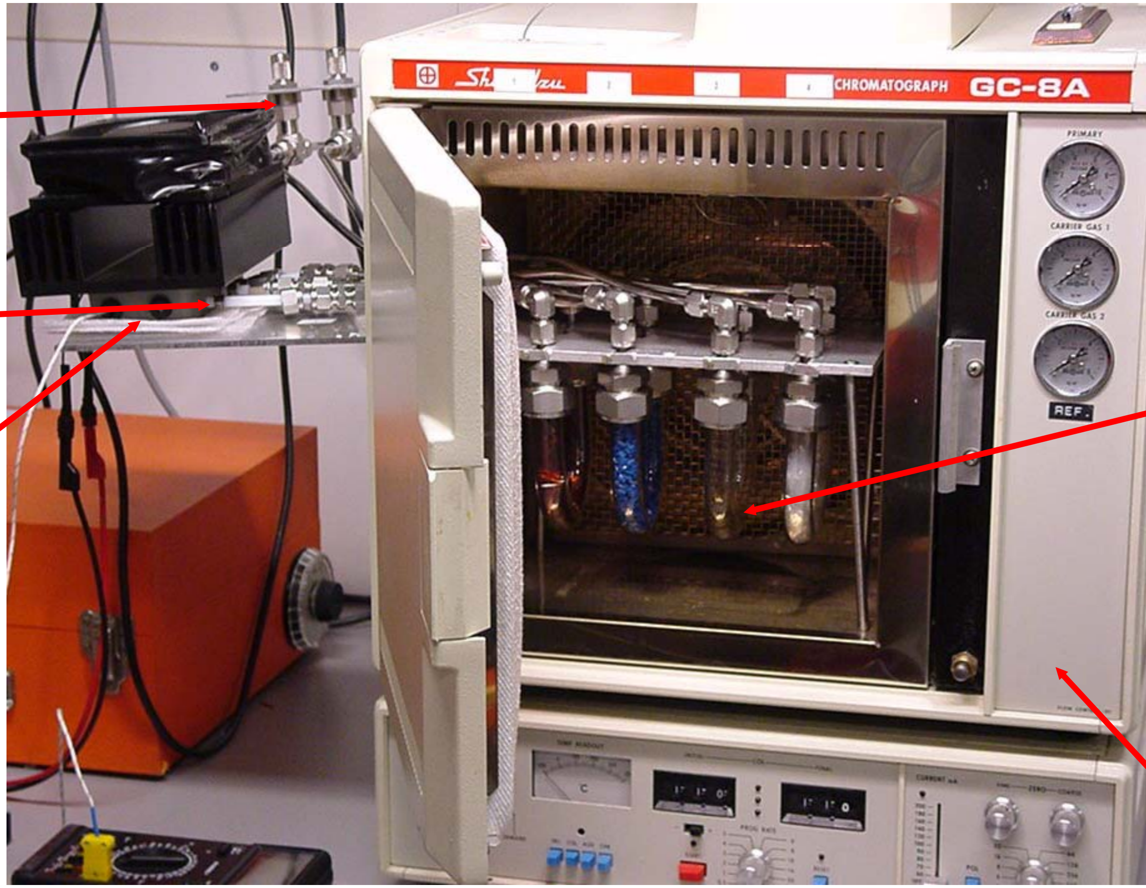
database of harmful *compounds*

Sampling + Analysis + Test -method

- weaknesses
  - –even more laborous and time consuming than the traditional one *in the beginning.*
  - –all compounds cannot be analyzed, some are not even commercially available.
  - –all gas mixtures cannot be analyzed (?)

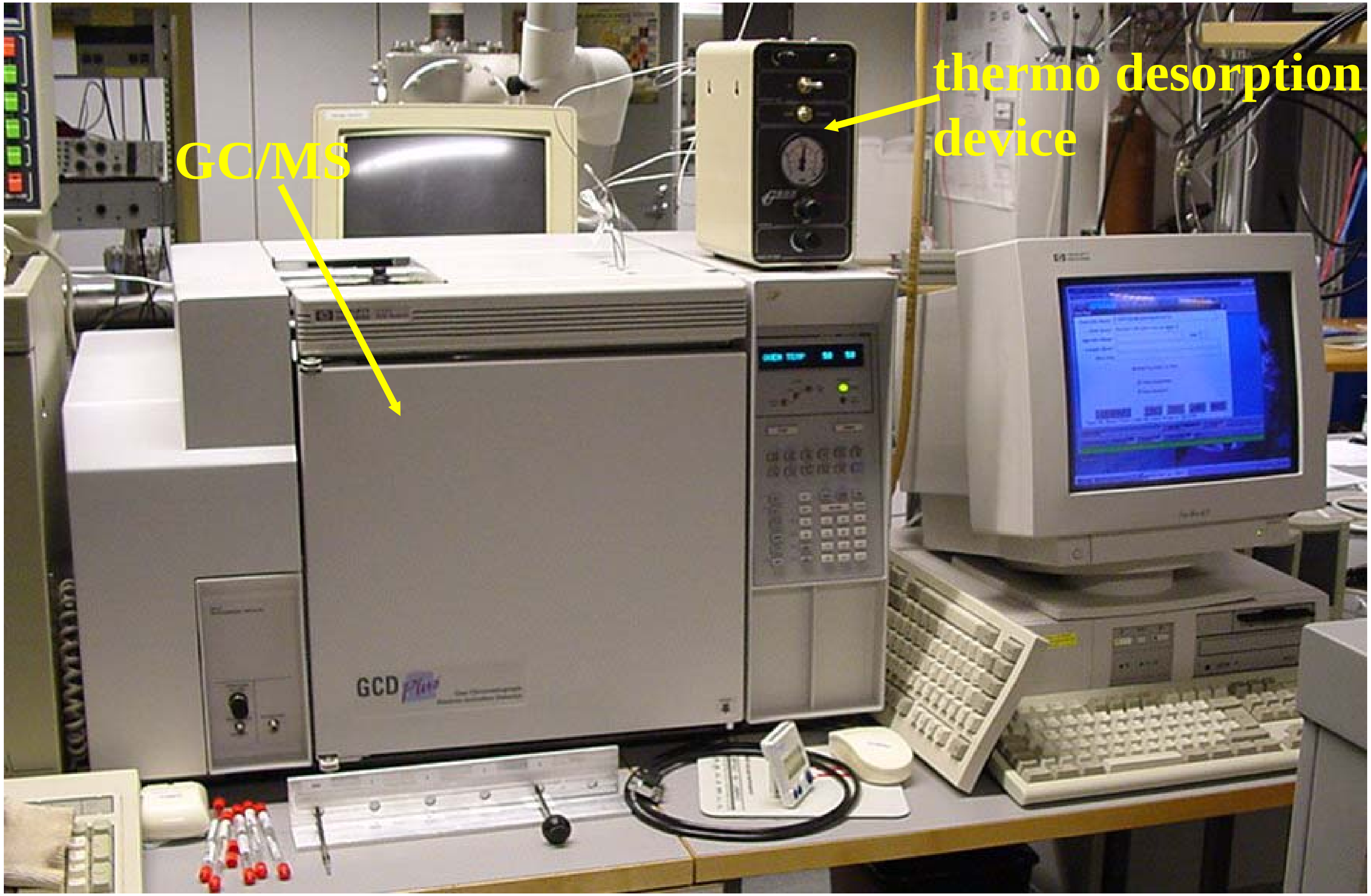
- benefits
  - – when the database is large enough, for introducing new materials only the sampling & analysis have to be performed (fast).
  - – sampling, analysis and test may be done in different sites (labs?).
  - – gas of a working detector may be monitoried (HEP experiments).
  - – gives some information about polymerisation processes in the detector.

Sampling with thermo desorption (TD) method



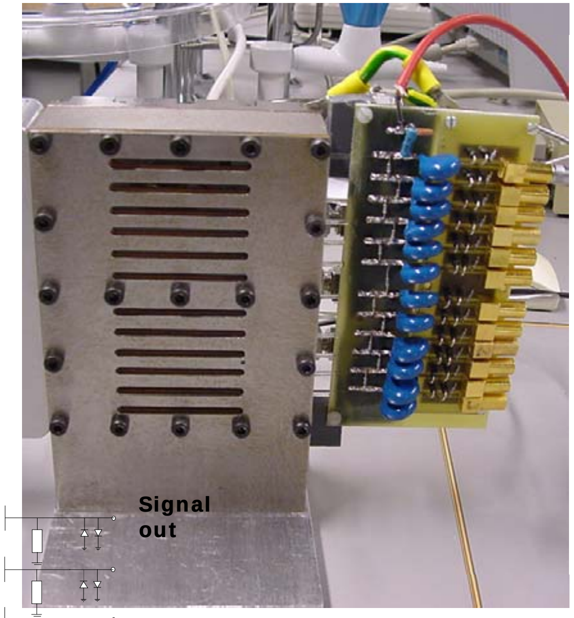
- Tenax TA used as an absorbant (range C5-C26)
- days/weeks long sampling time possible -> good sensitivity

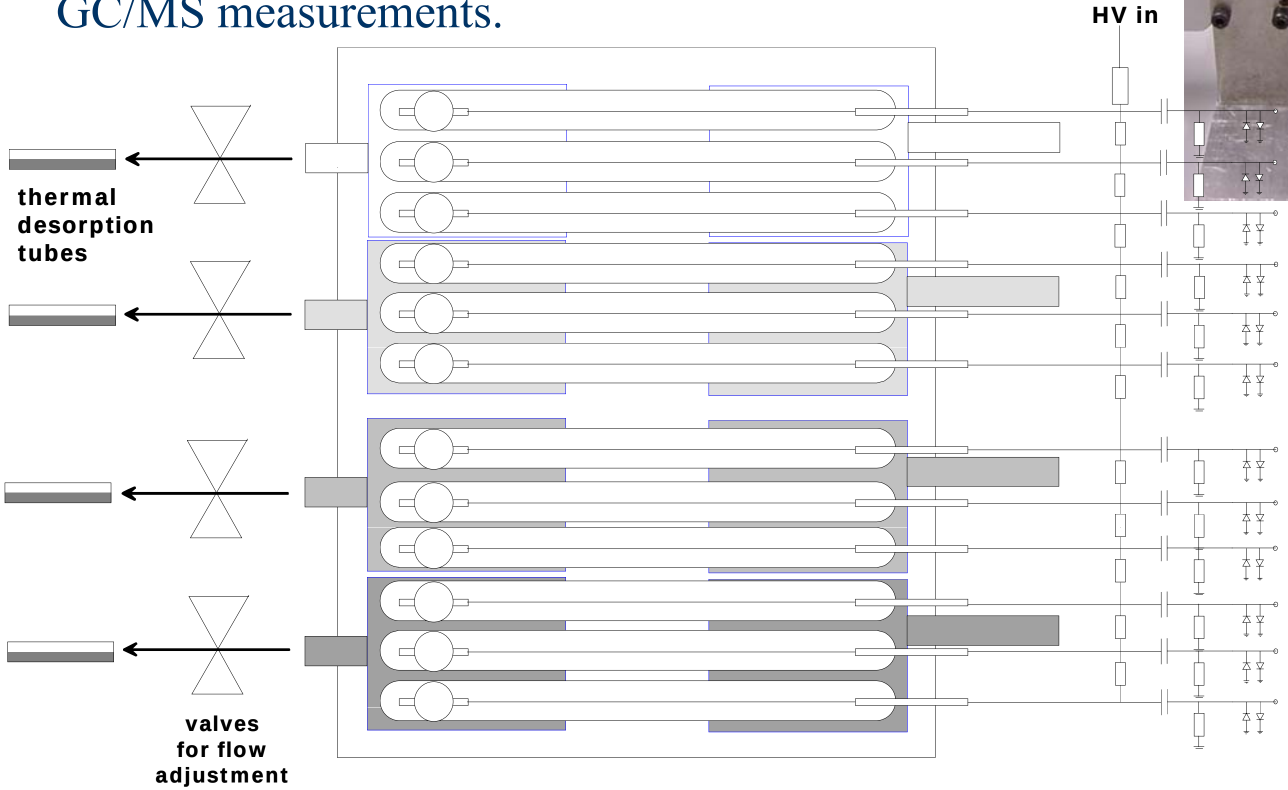
GC/MS with a TD device



Irradiation chamber of 12 proportional counters

• gas flow divided into four separate sections for inclusion of different impurities (3 detectors/section)
• impurity monitored during the irradiation by regular GC/MS measurements.

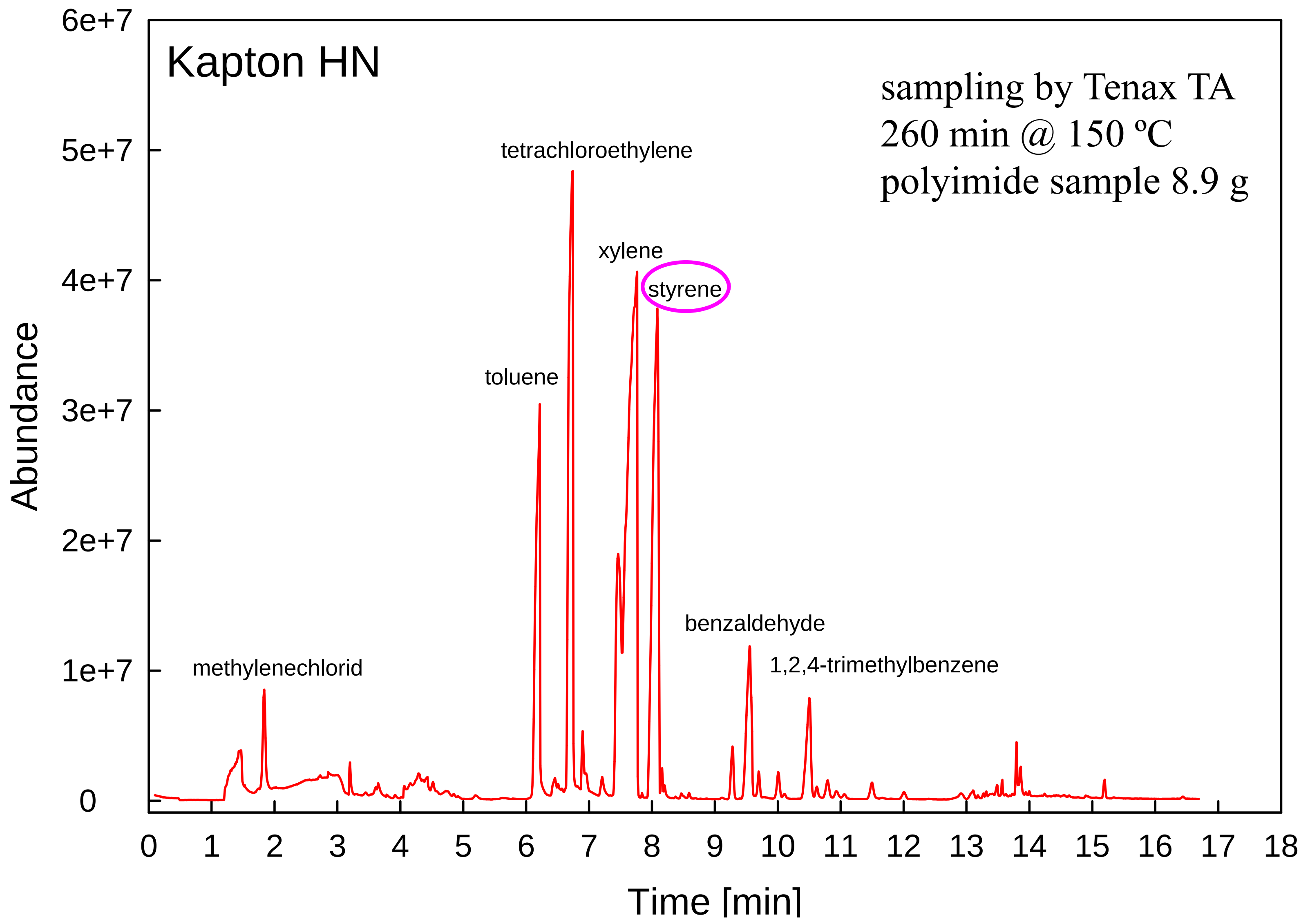




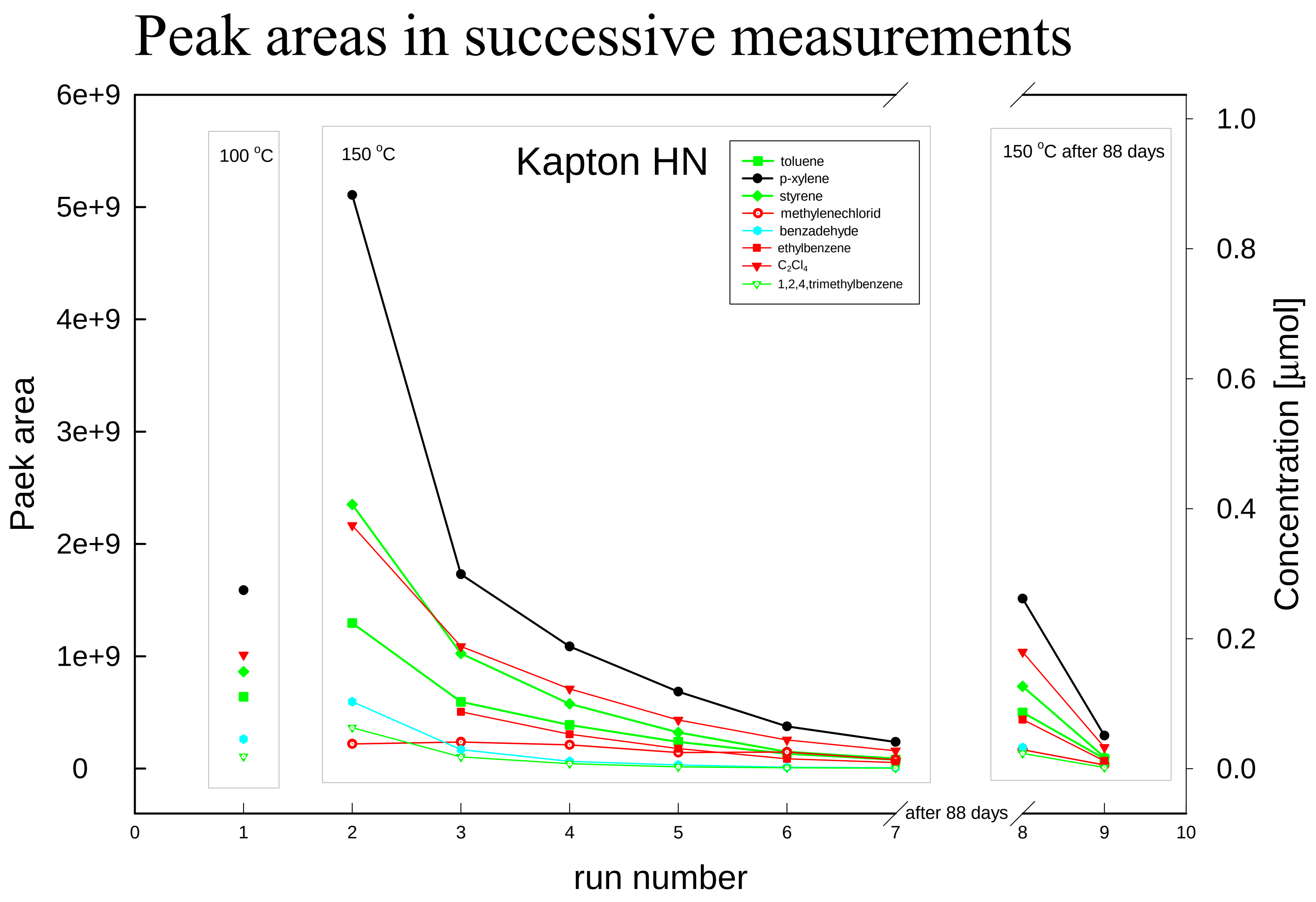
irradiation
by X-ray device
(Cu-target)

monitoring
by ^{55}Fe X-ray
source

Results - Outgassing Analysis

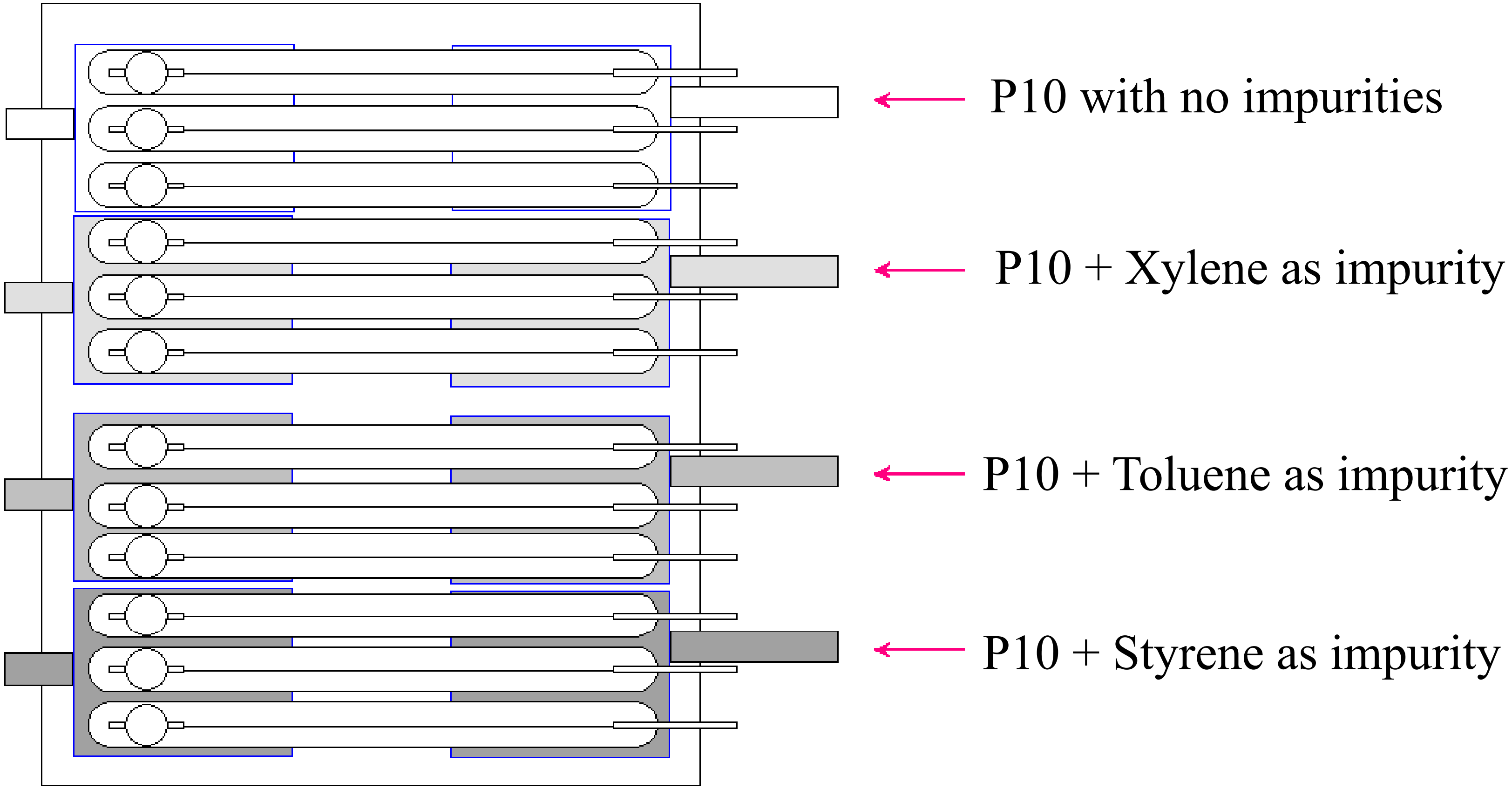


Results - Outgassing Analysis

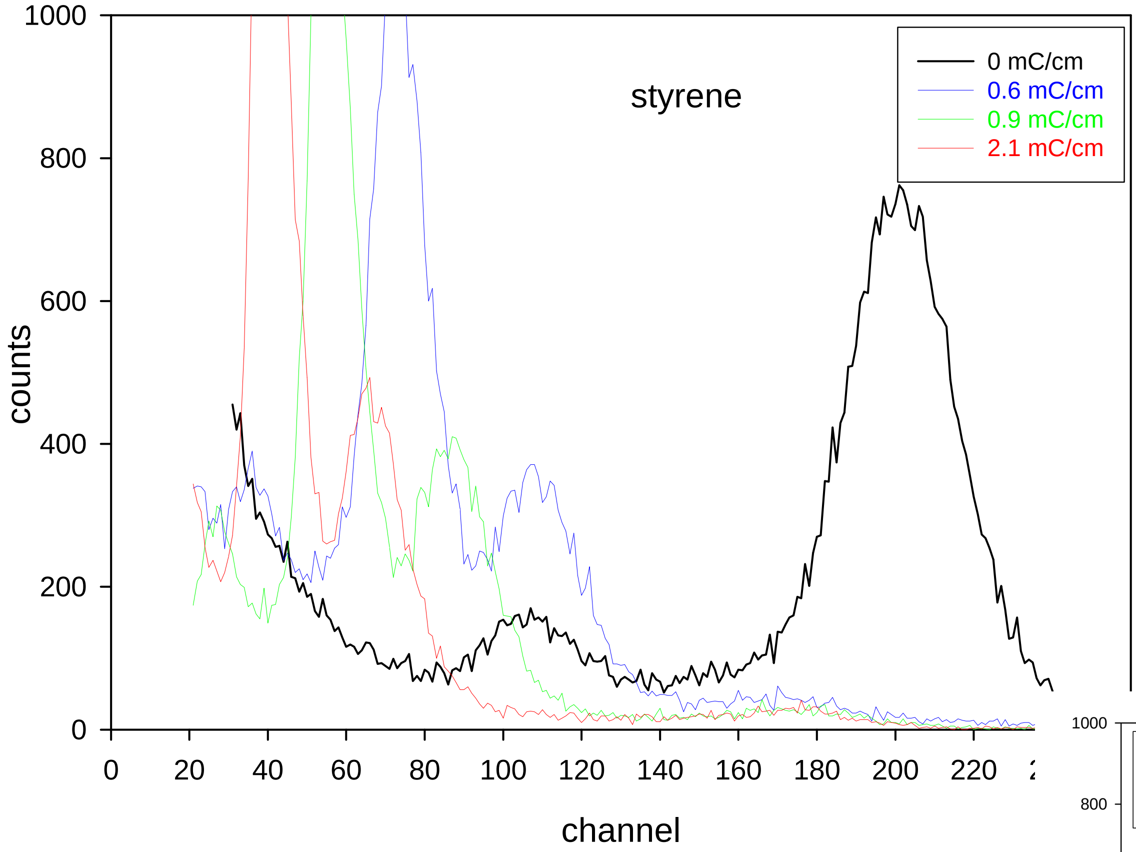


Test setup

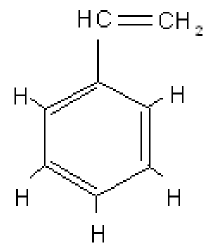
Accelerated aging test with aromatic solvents

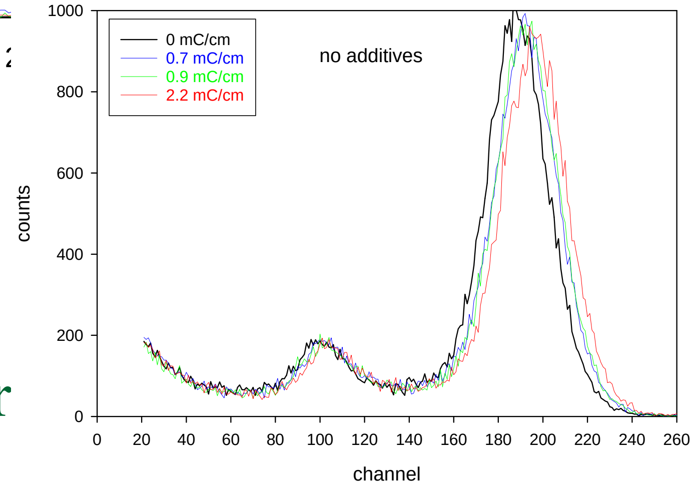


Results - Accelerated Aging Test



STYRENE
added to P-10
gas mixture

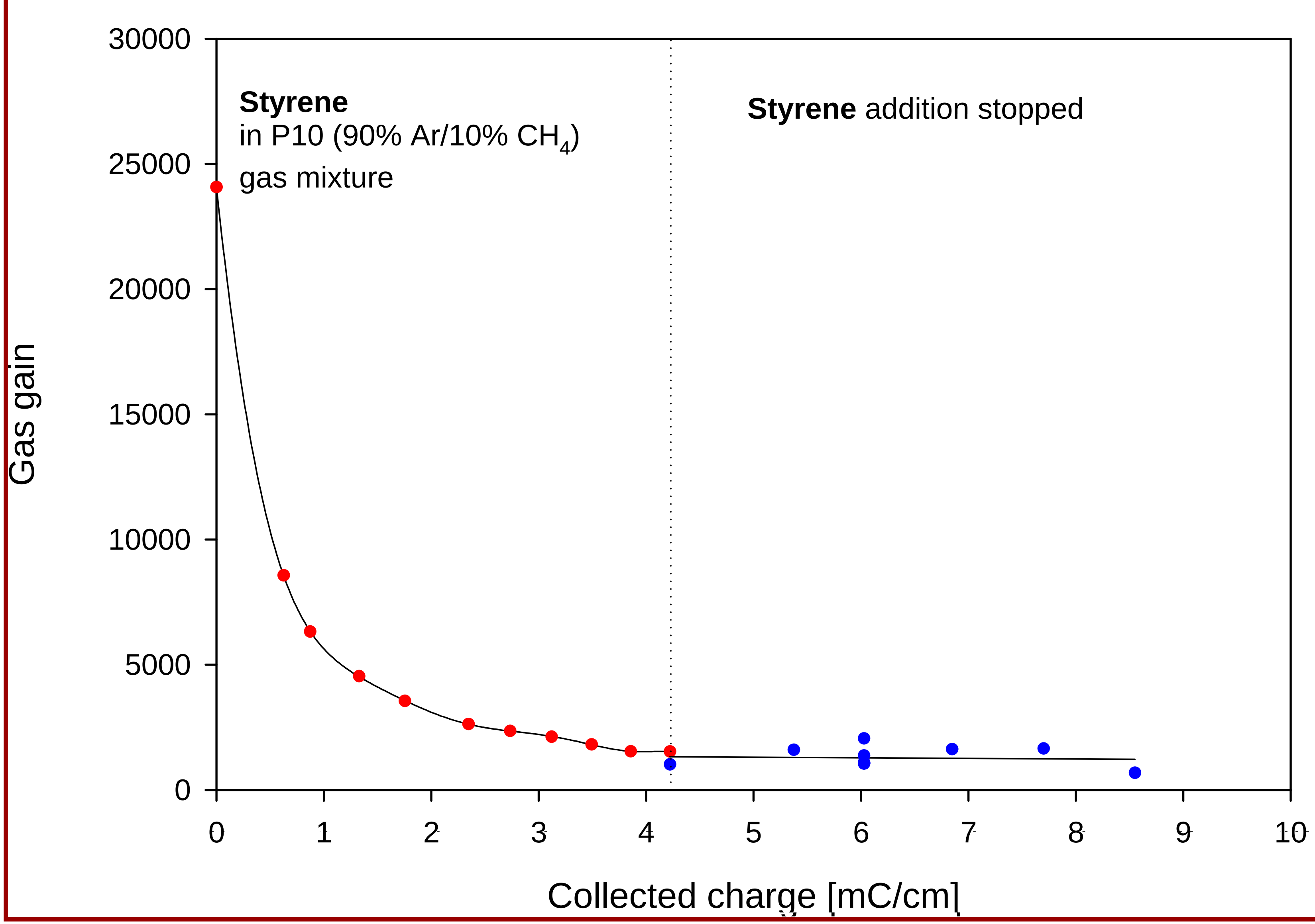




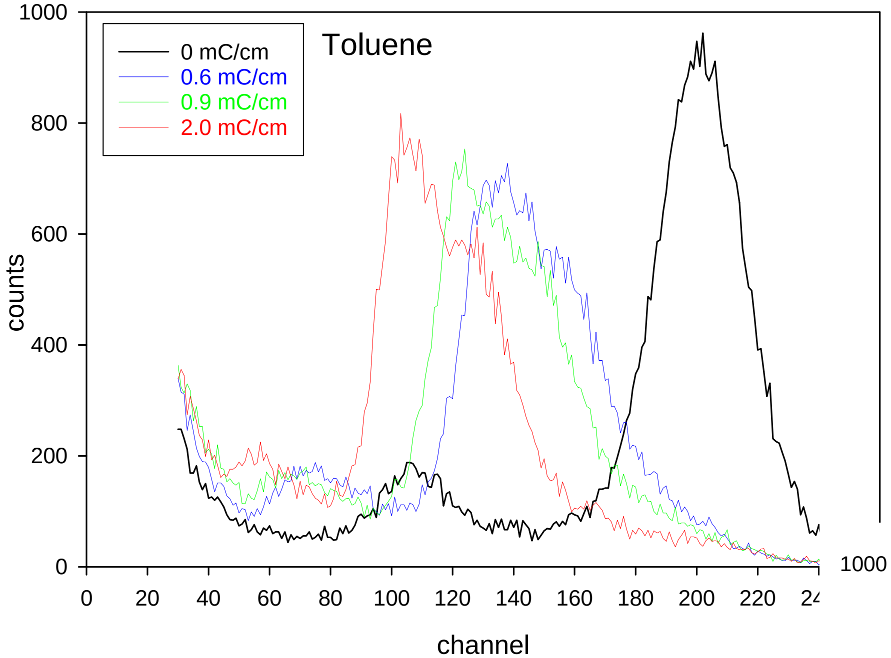
X-rays: 7 kVp, 190 μA
Detector current: < 100 nA/(exposed wire cm)

the reference detector

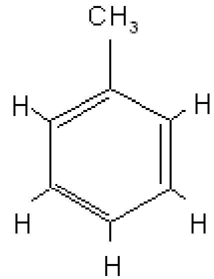
Results - Accelerated Aging Test

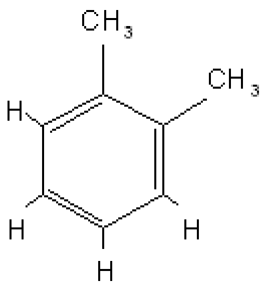


Results - Accelerated Aging Test

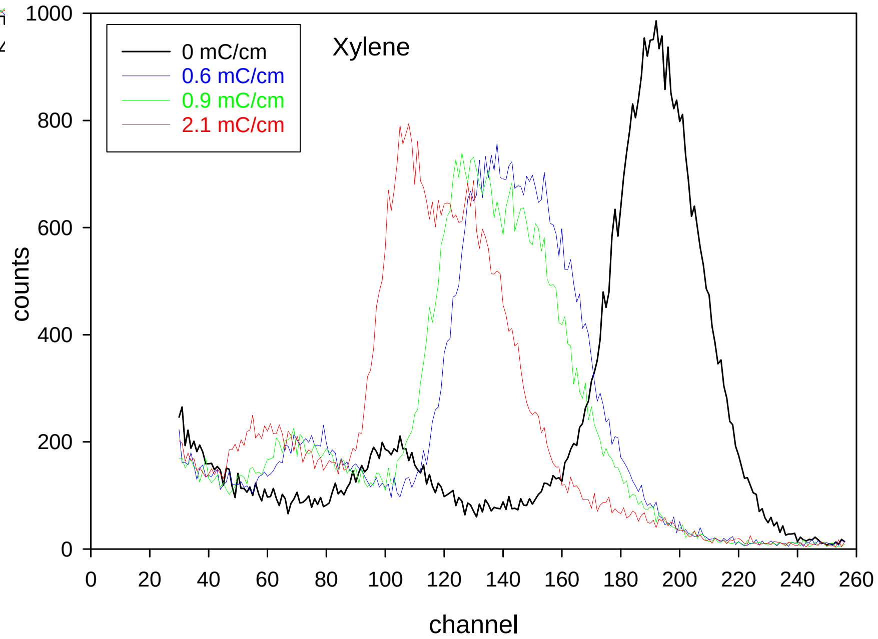


Impurity:
TOLUENE

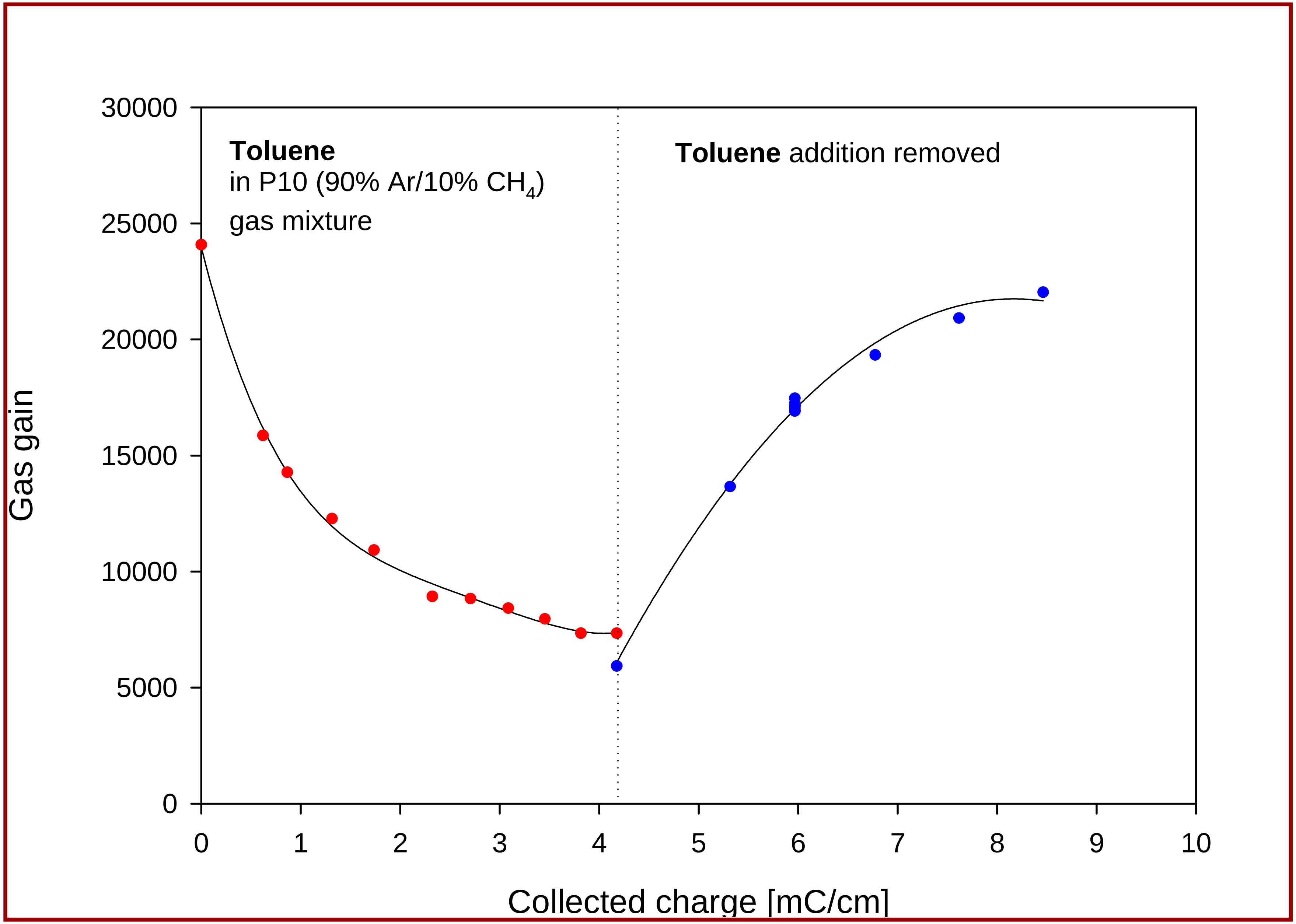




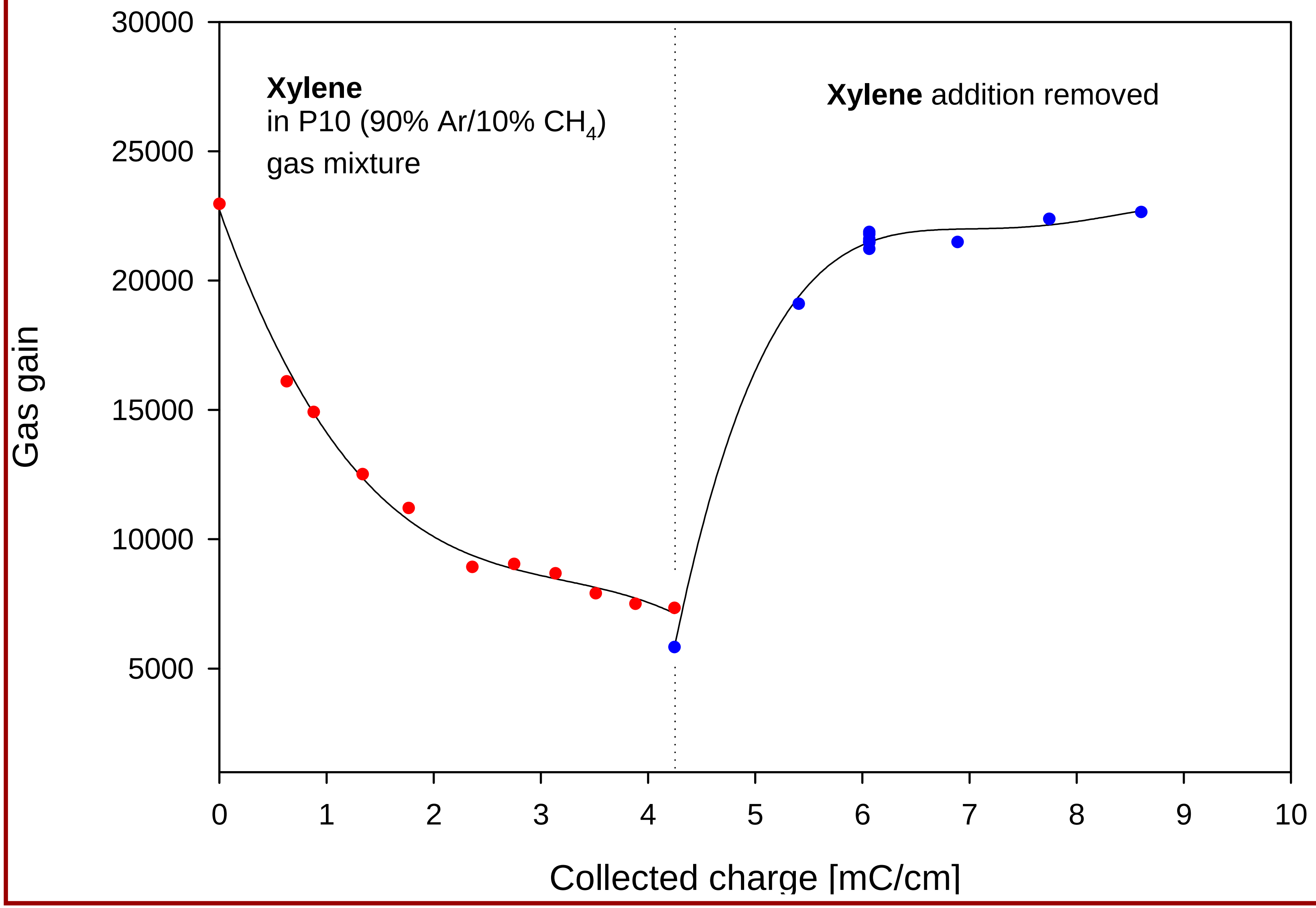
Impurity:
XYLENE
added



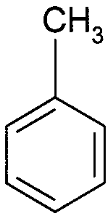
Results - Accelerated Aging Test



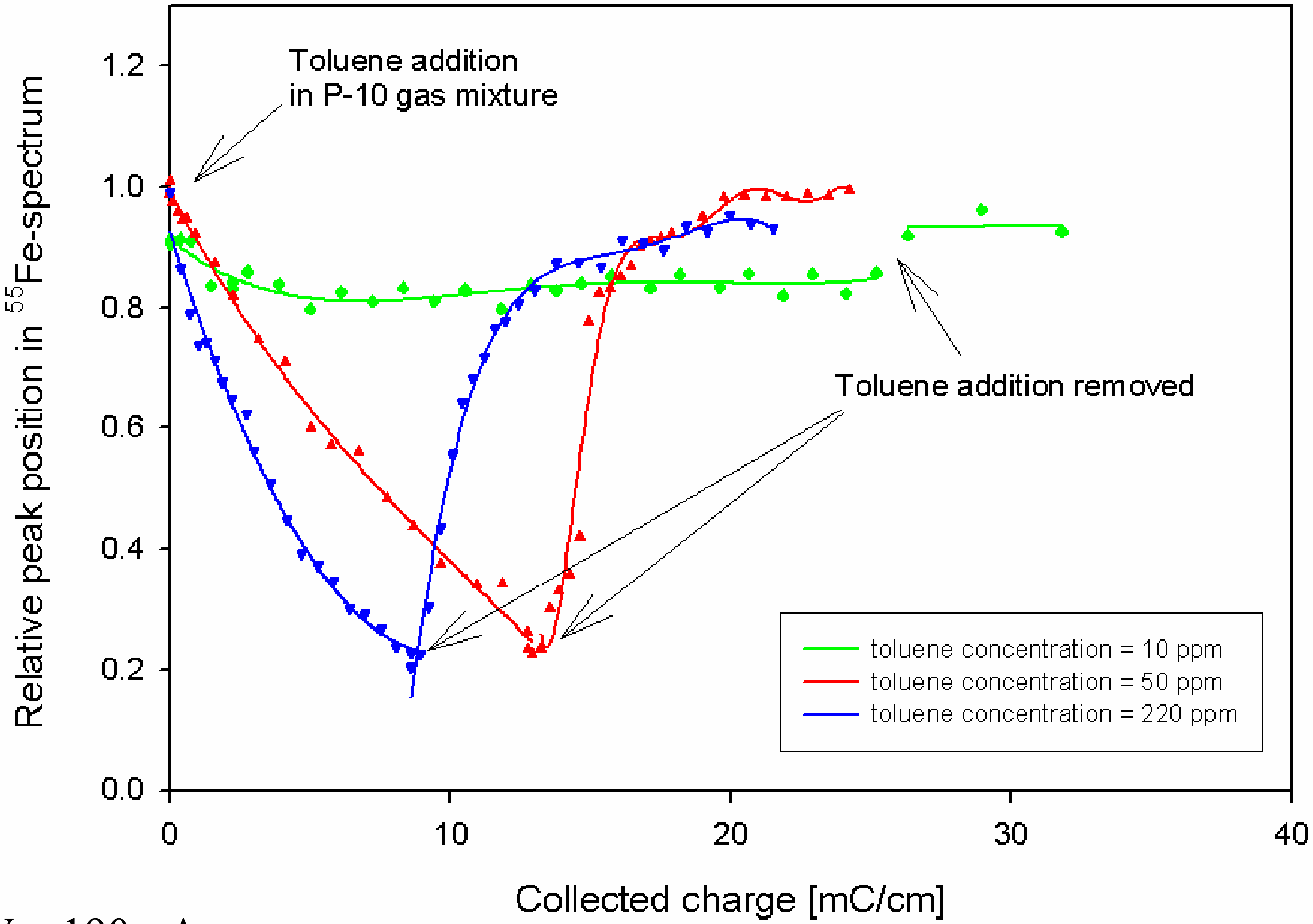
Results - Accelerated Aging Test



Results - Accelerated Aging Test

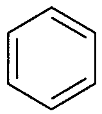


Aging of a proportional counter
with different impurities of toluene in P-10 gas mixture

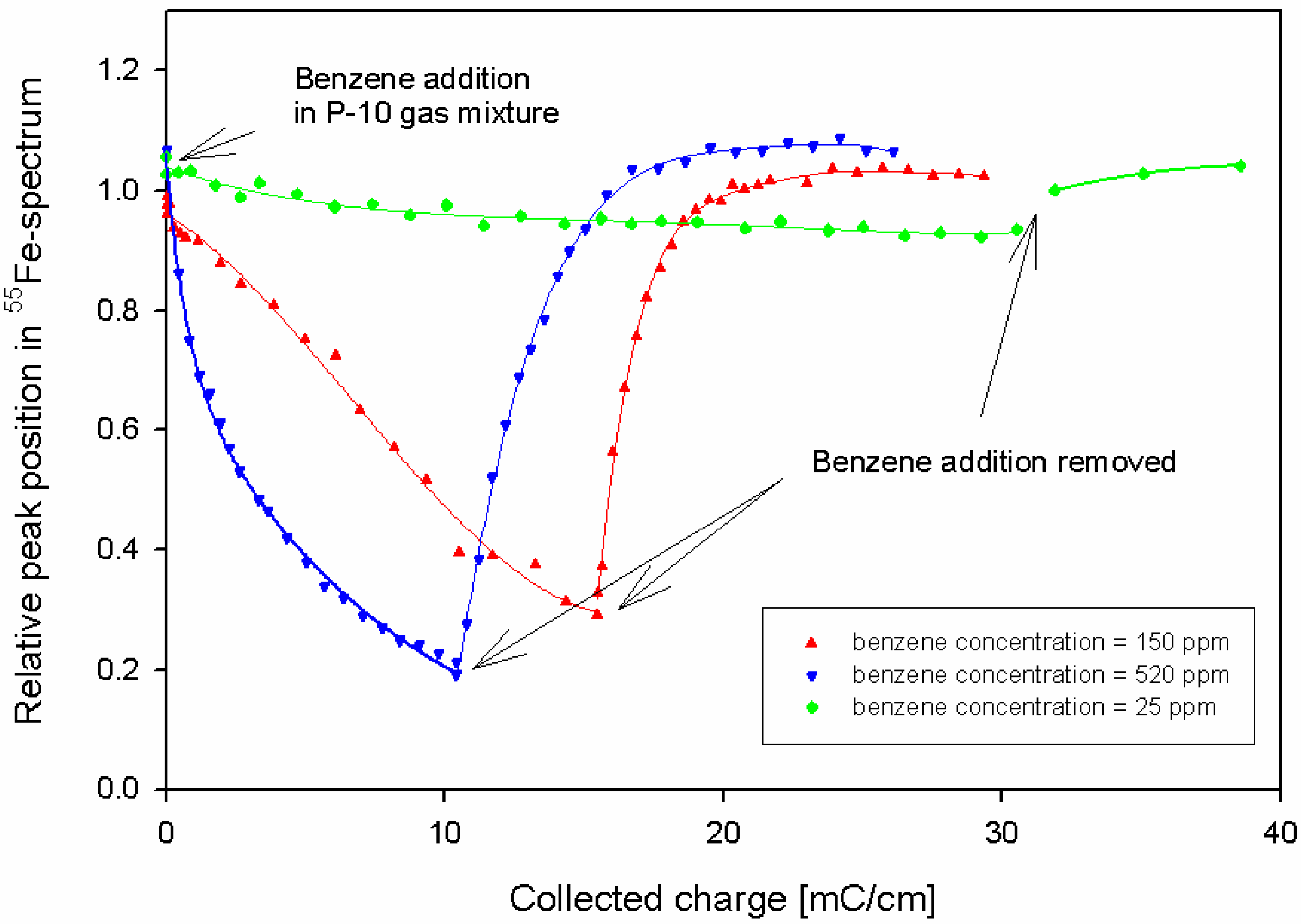


X-rays: 7 kVp, 190 μA
Detector current: < 100 nA/(exposed wire cm)

Results - Accelerated Aging Test

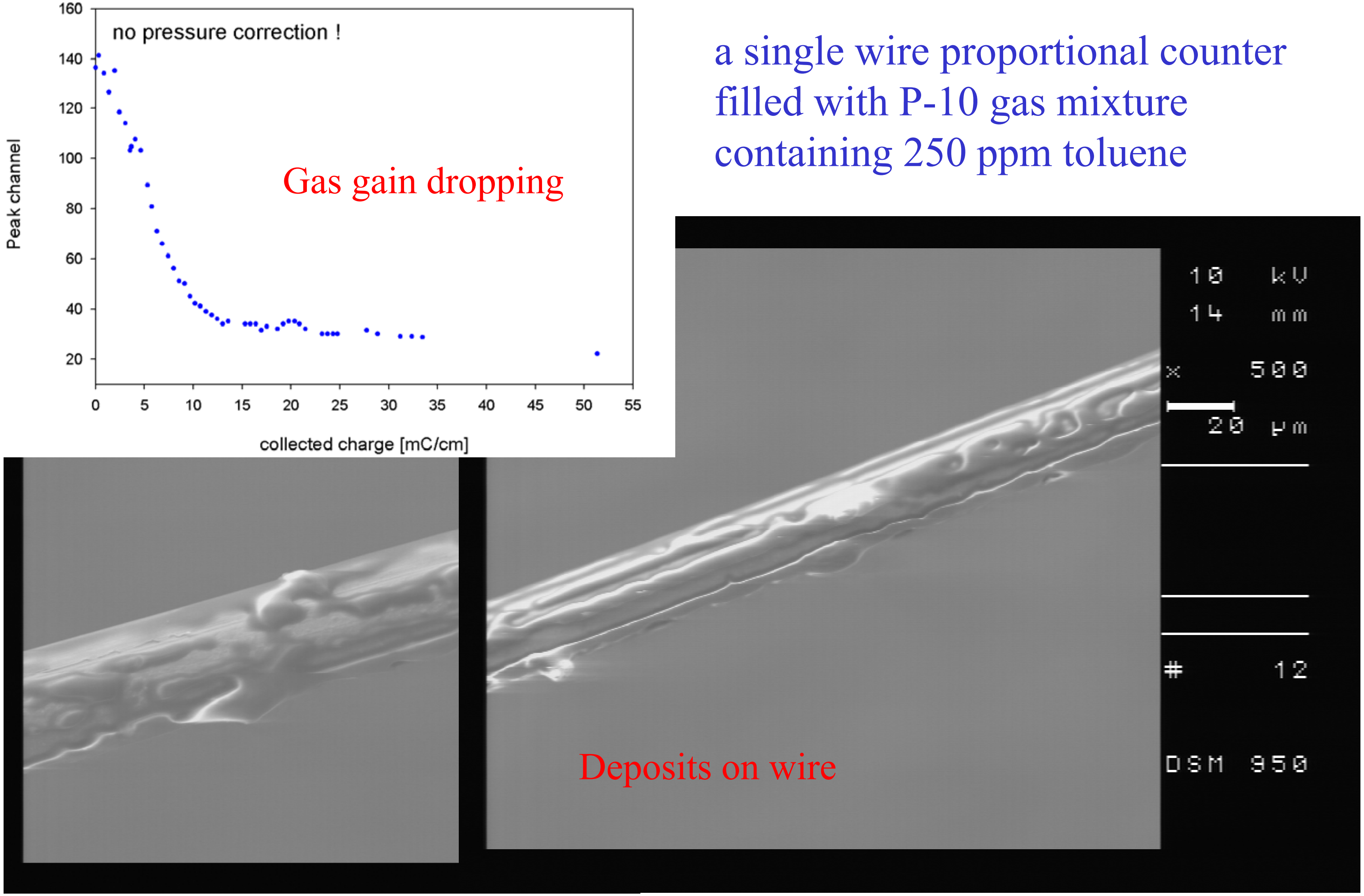


Aging of a proportional counter with different impurities of benzene in P-10 gas mixture



Confirmation of ageing with a different detector and a source

relative gas amplification during irradiation by ^{244}Am source



a single wire proportional counter filled with P-10 gas mixture containing 250 ppm toluene

H.Yasuda: Plasma Polymerization:

CHAPTER 7

Competitive Ablation and Polymer Formation in Plasma

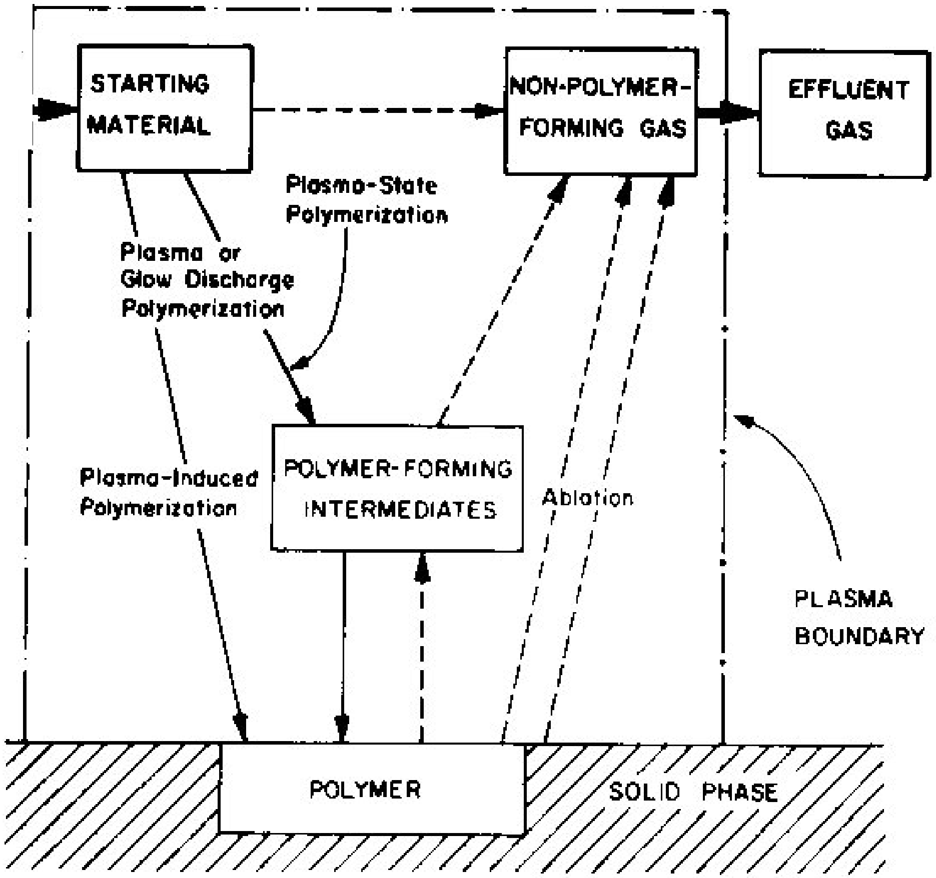
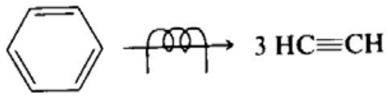


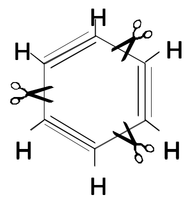
Fig. 7.2 Overall mechanism of glow discharge polymerization.

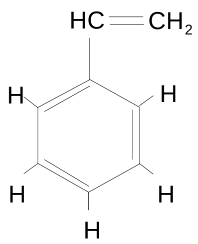
H.Yasuda: Plasma Polymerization:

6.4.4.1 AROMATIC STRUCTURE

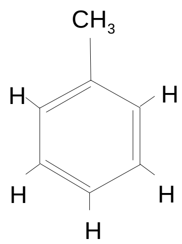
Acetylene (HC≡CH) and benzene (⌬) are very similar in their plasma polymerization characteristics. Both compounds form plasma polymers with the least amount of hydrogen production (group I monomer), and their characteristics of copolymerization with N_2 and/or H_2O are nearly identical if we consider that one molecule of benzene is equivalent to three molecules of acetylene:



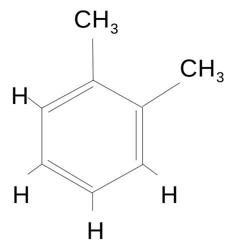




STYRENE



TOLUENE

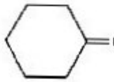
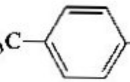


XYLENE

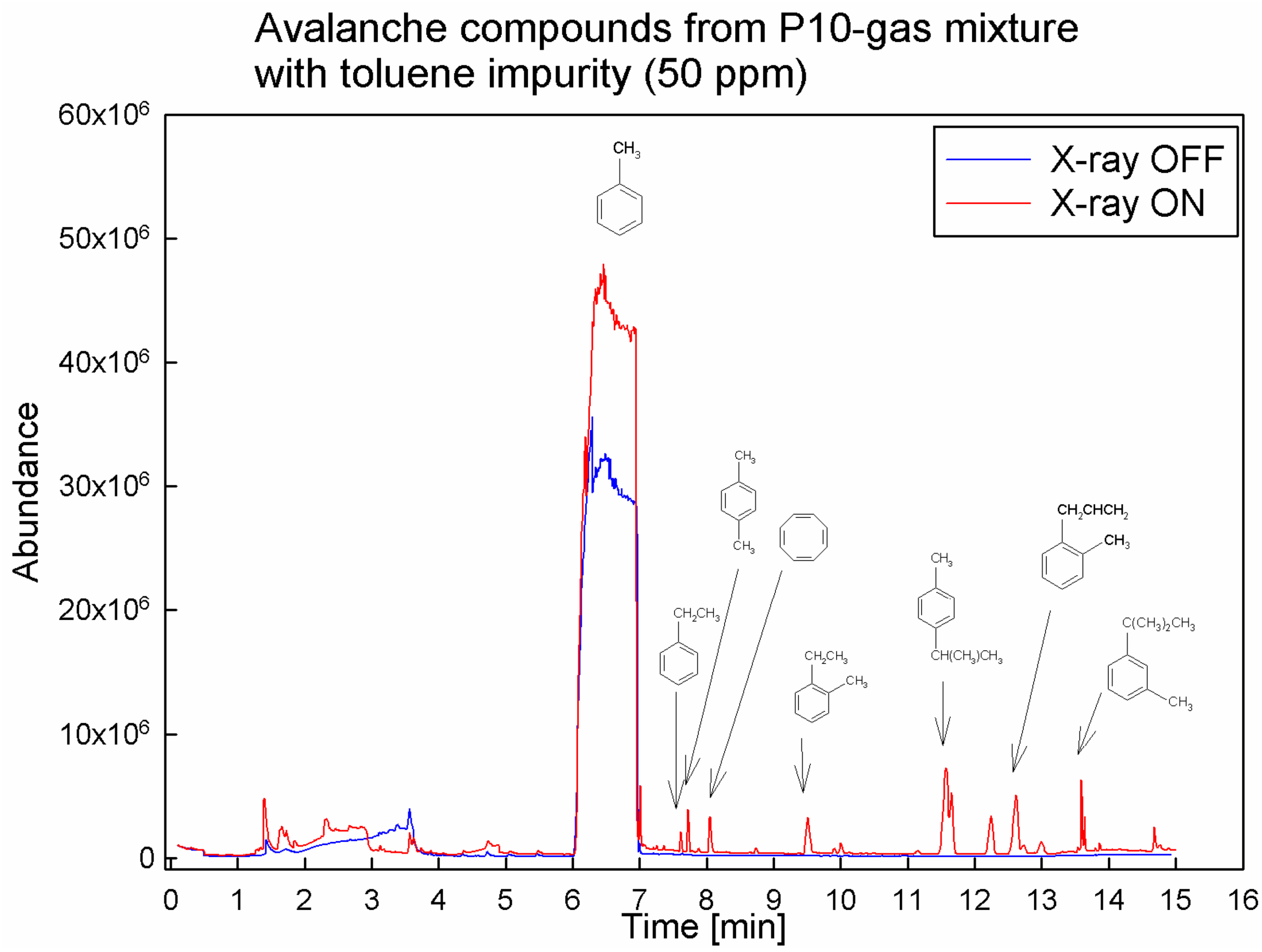
H.Yasuda: Plasma Polymerization:

Table 8.8

HYDROCARBON POLYMERIZATION PARAMETERS[a]

Compound	Structure	$t_{1/2}$ (sec)	Monomer-type parameter γ	Fraction of residual vapor x	Hydrogen yield y (ratio)	Polymer yield $(1 - x)$
Cycloheptane		3.5	1.69	0.083	1.61	0.91
Cyclohexene		2	0.833	0.033	0.800	0.96
Methylenecyclohexane	 $=CH_2$	—[b]	1.01	0.026	0.979	0.97
Benzene		<2	0.110	0.003	0.107	0.99
Toluene	 $-CH_3$	3	0.174	0.001	0.172	9.90
p-Xylene	H_3C-  $-CH_3$	4	0.133	0.000	0.133	1.00
Ethylbenzene	 $-CH_2CH_3$	6	0.298	0.020	0.278	0.98
Styrene	 $-CH{=}CH_2$	4	0.105	0.017	0.088	0.98

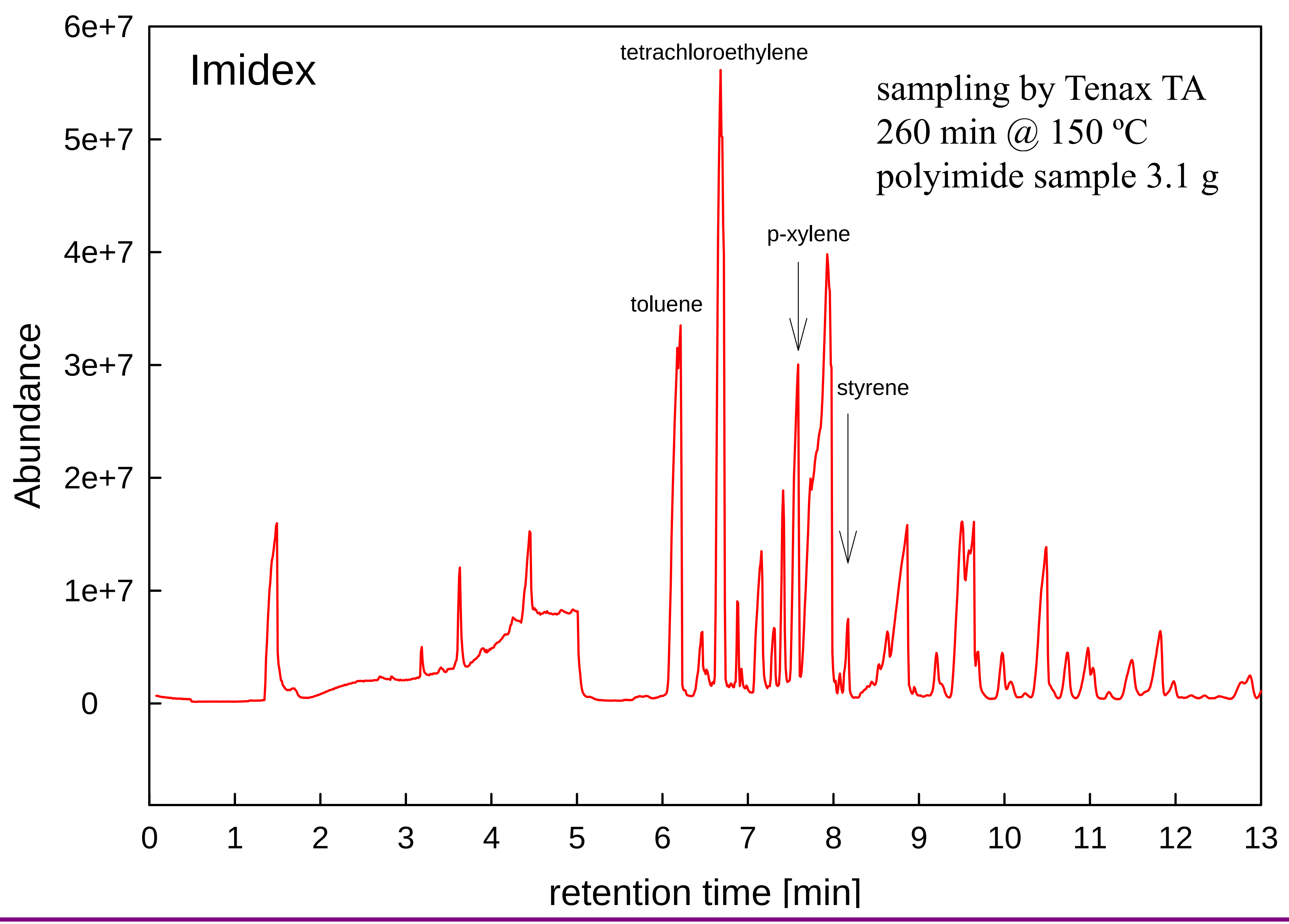
Results – Analysis of avalanche compounds



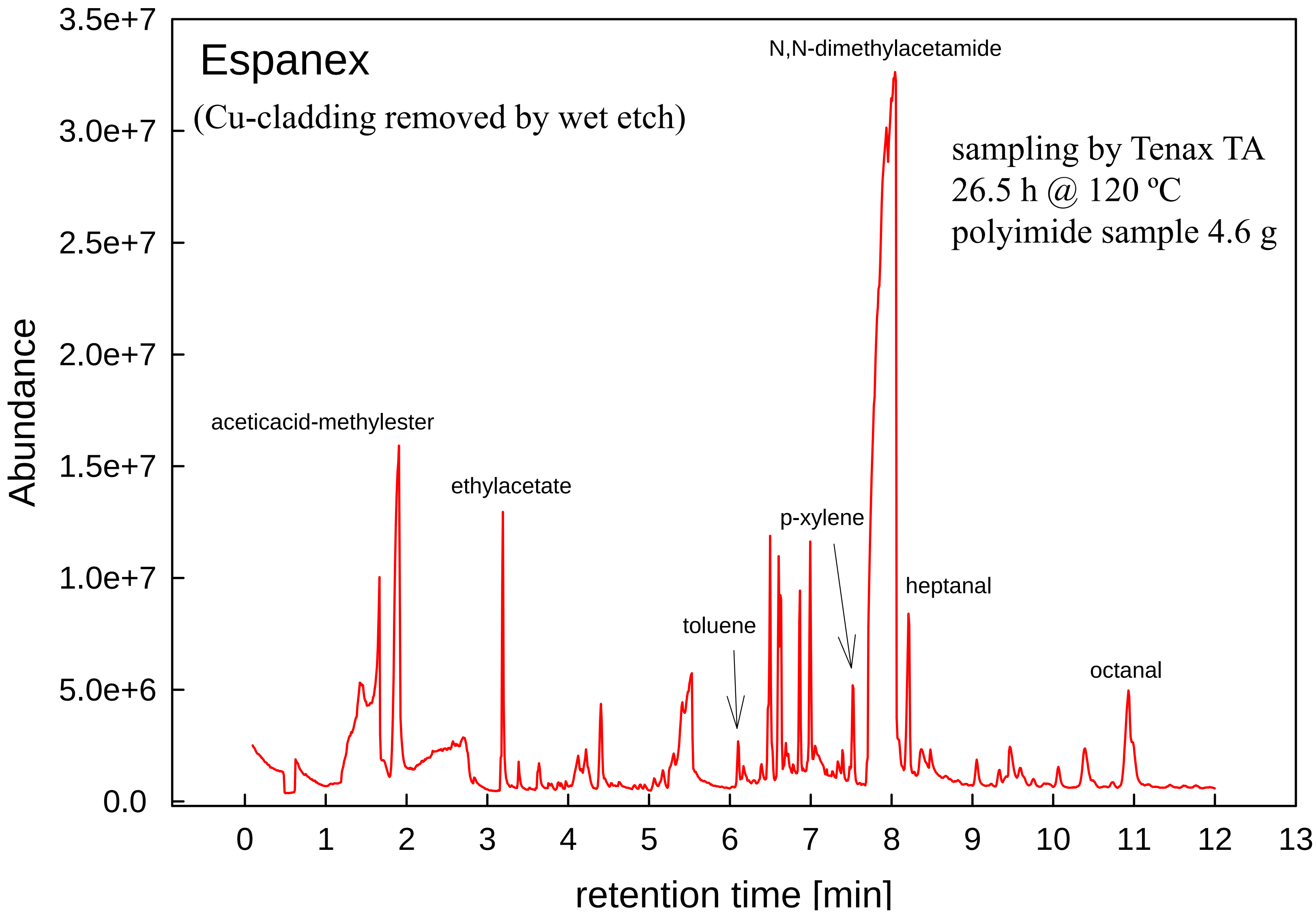
Outgassing analysis of some common detector materials

analyzed:
- polyimides (4 different grades)
- PET, PEEK, PA
- FR4
- soldering tin
- epoxies & glues
- cable insulating materials
- rubbers

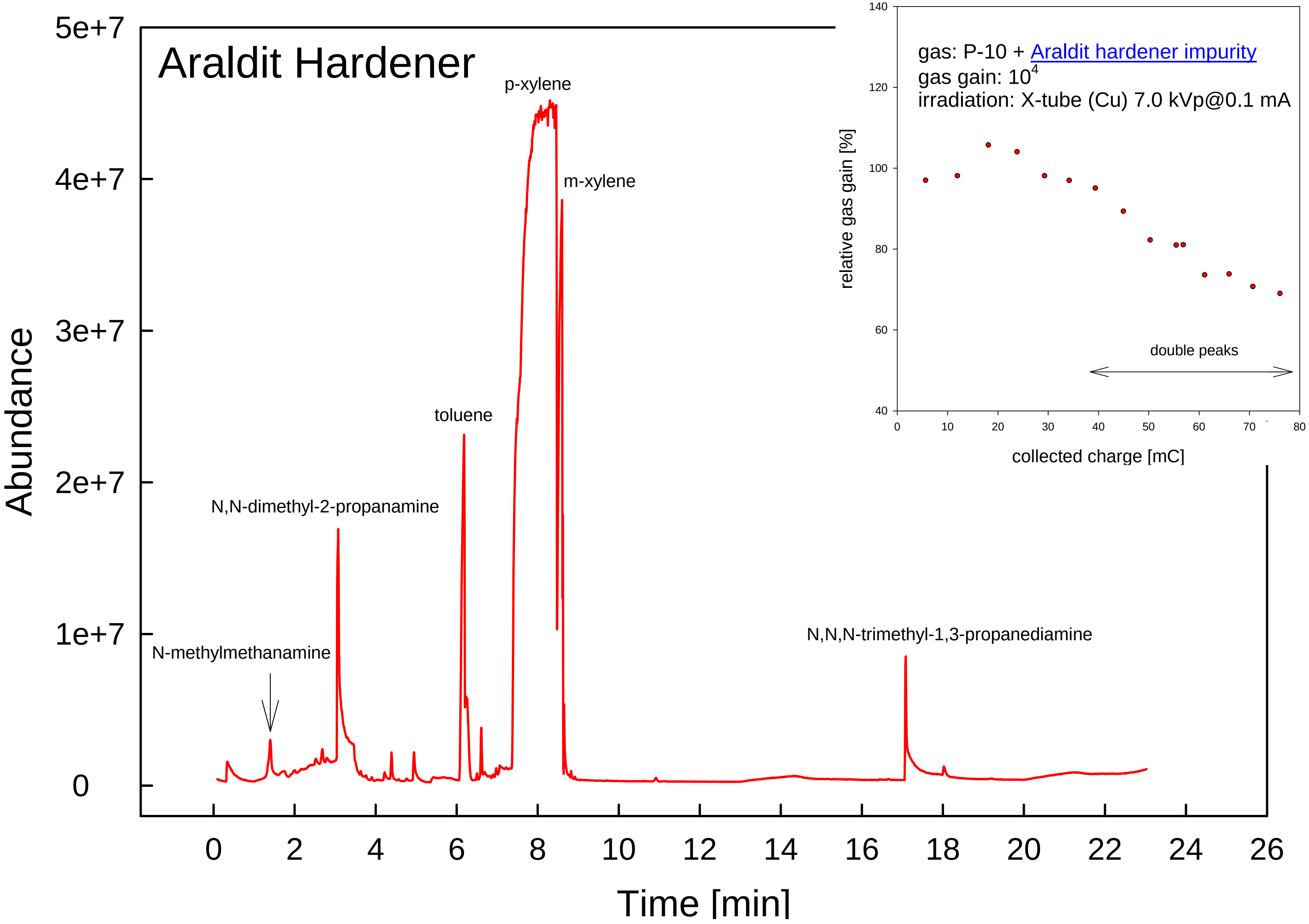
Results - Outgassing Analysis



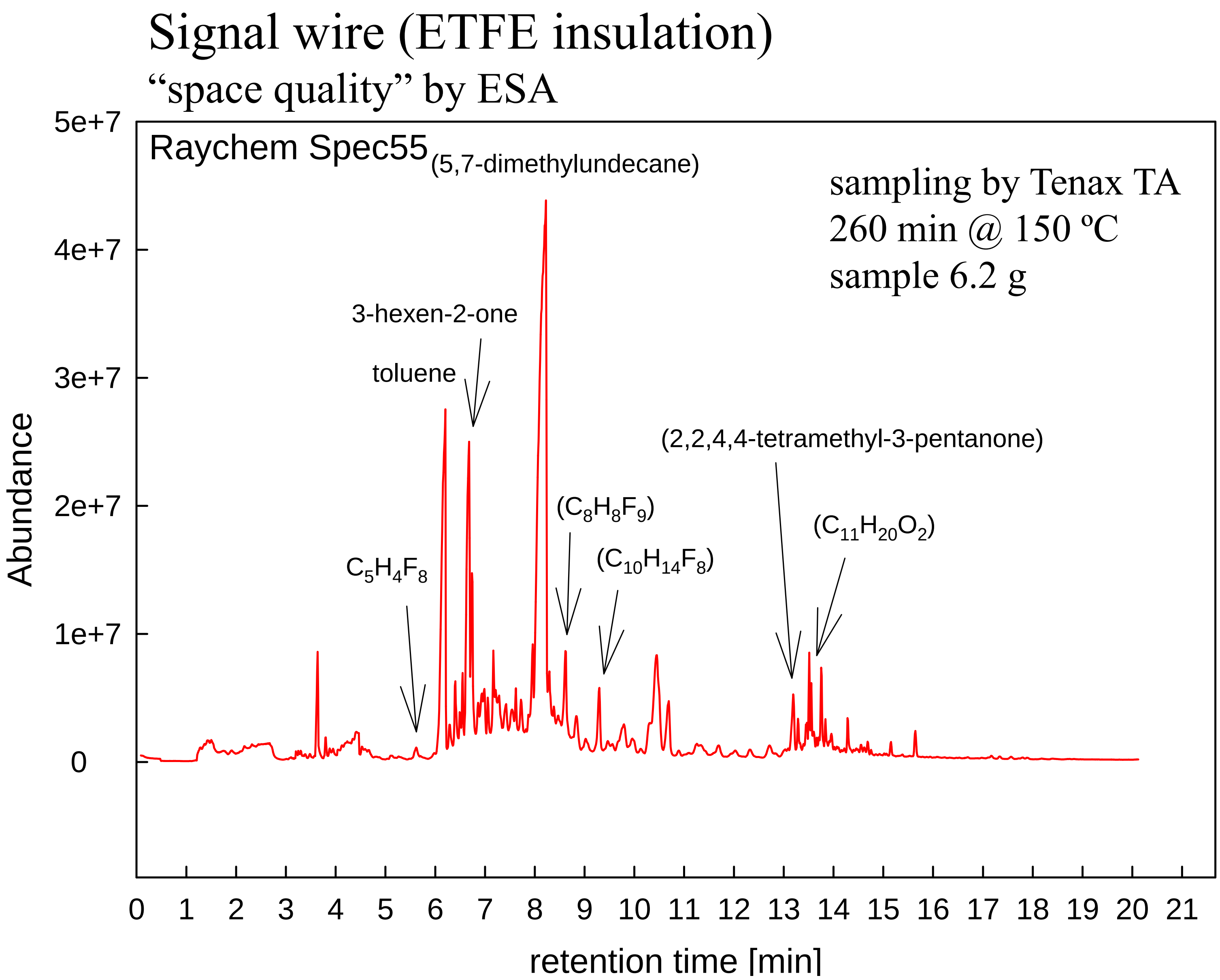
Results - Outgassing Analysis



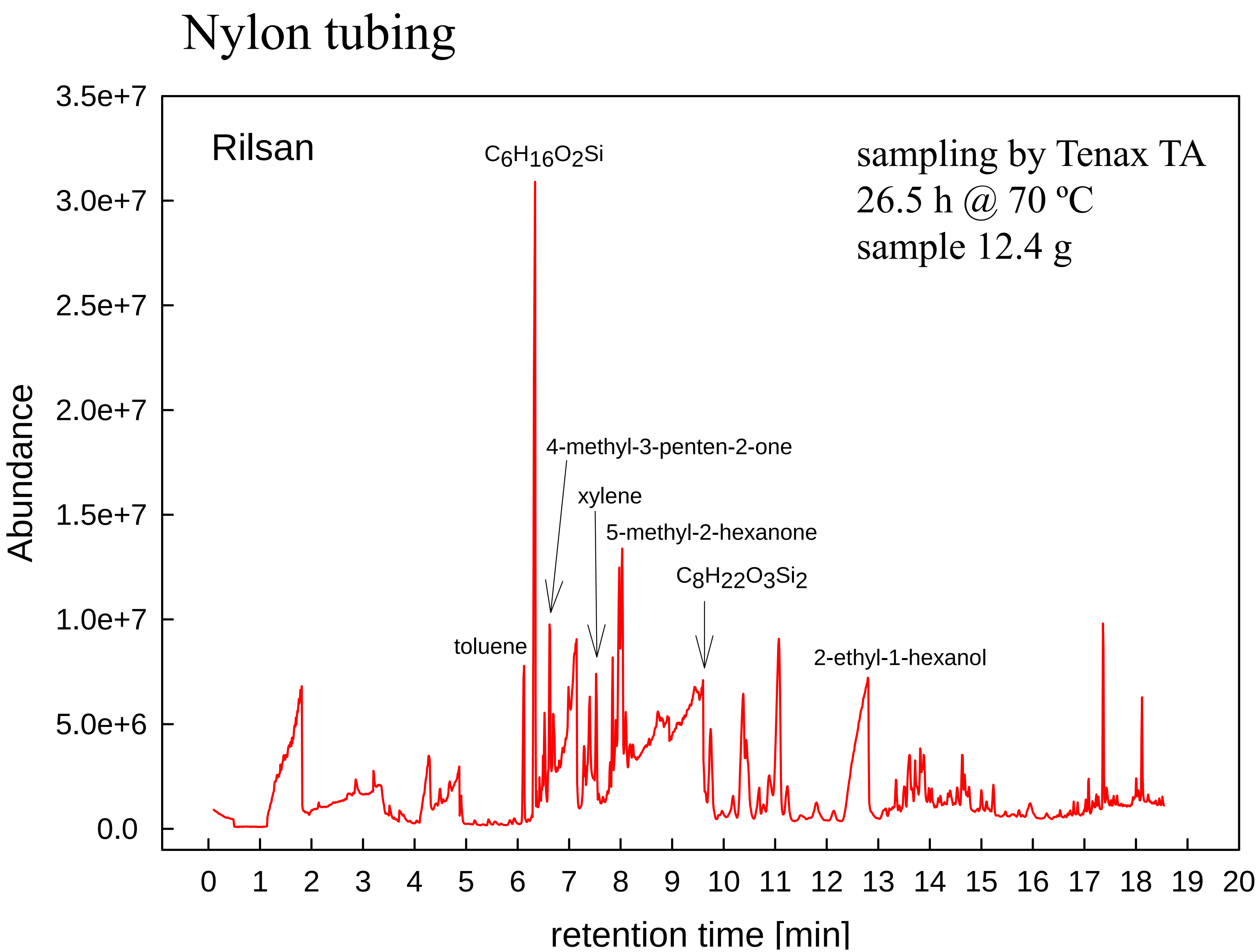
Results - Outgassing Analysis



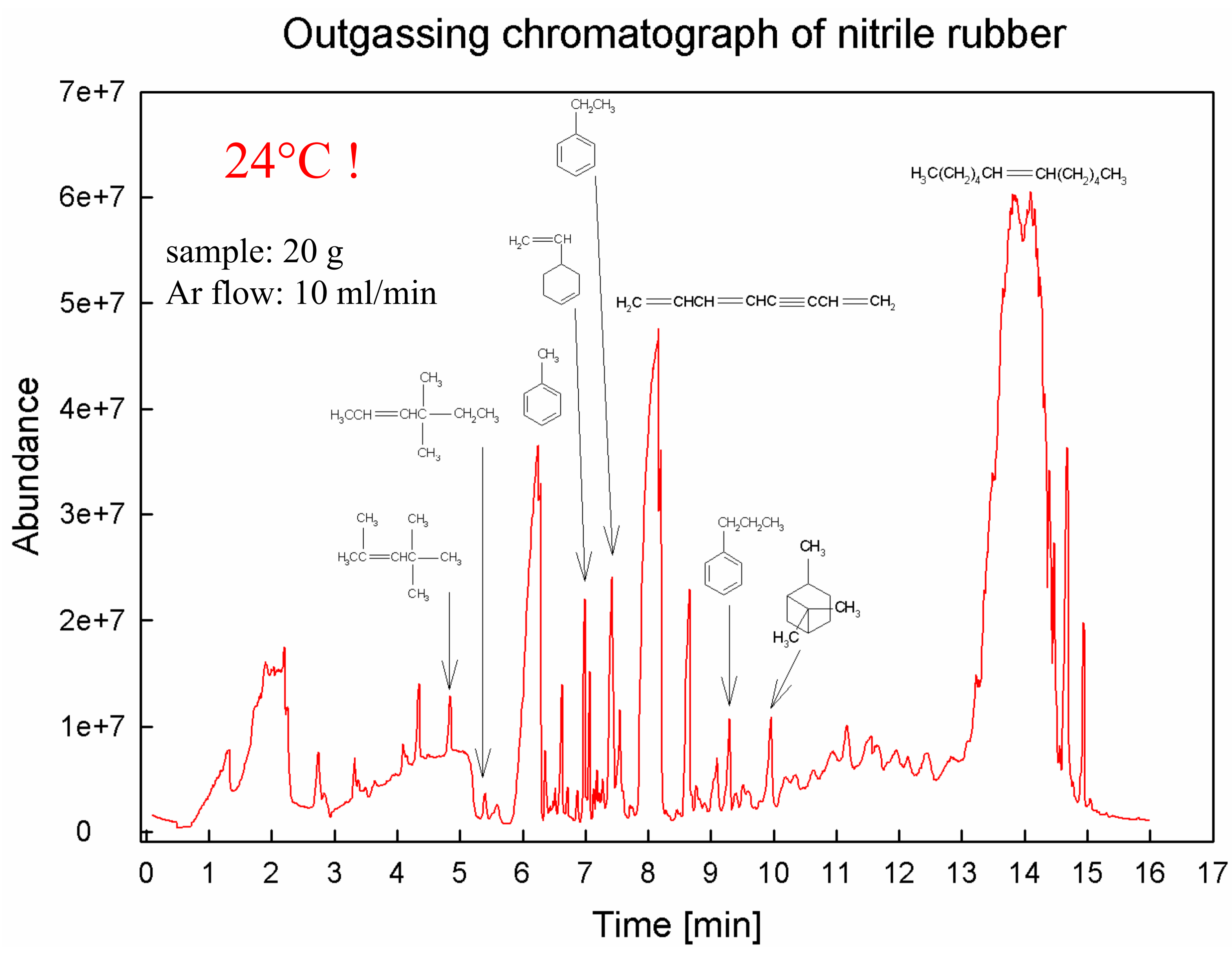
Results - Outgassing Analysis



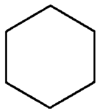
Results - Outgassing Analysis



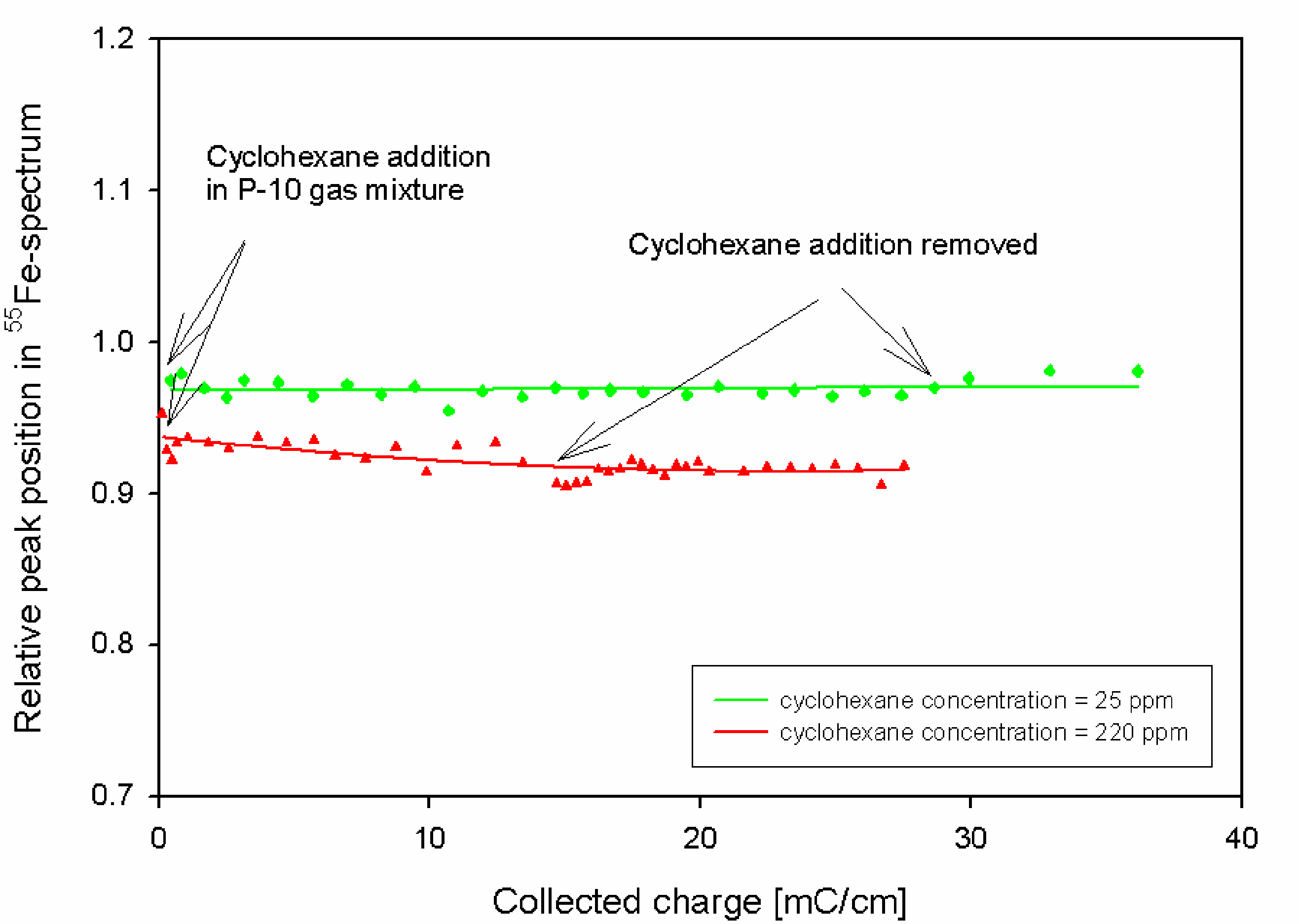
Results - Outgassing Analysis



Results - Accelerated Aging Test

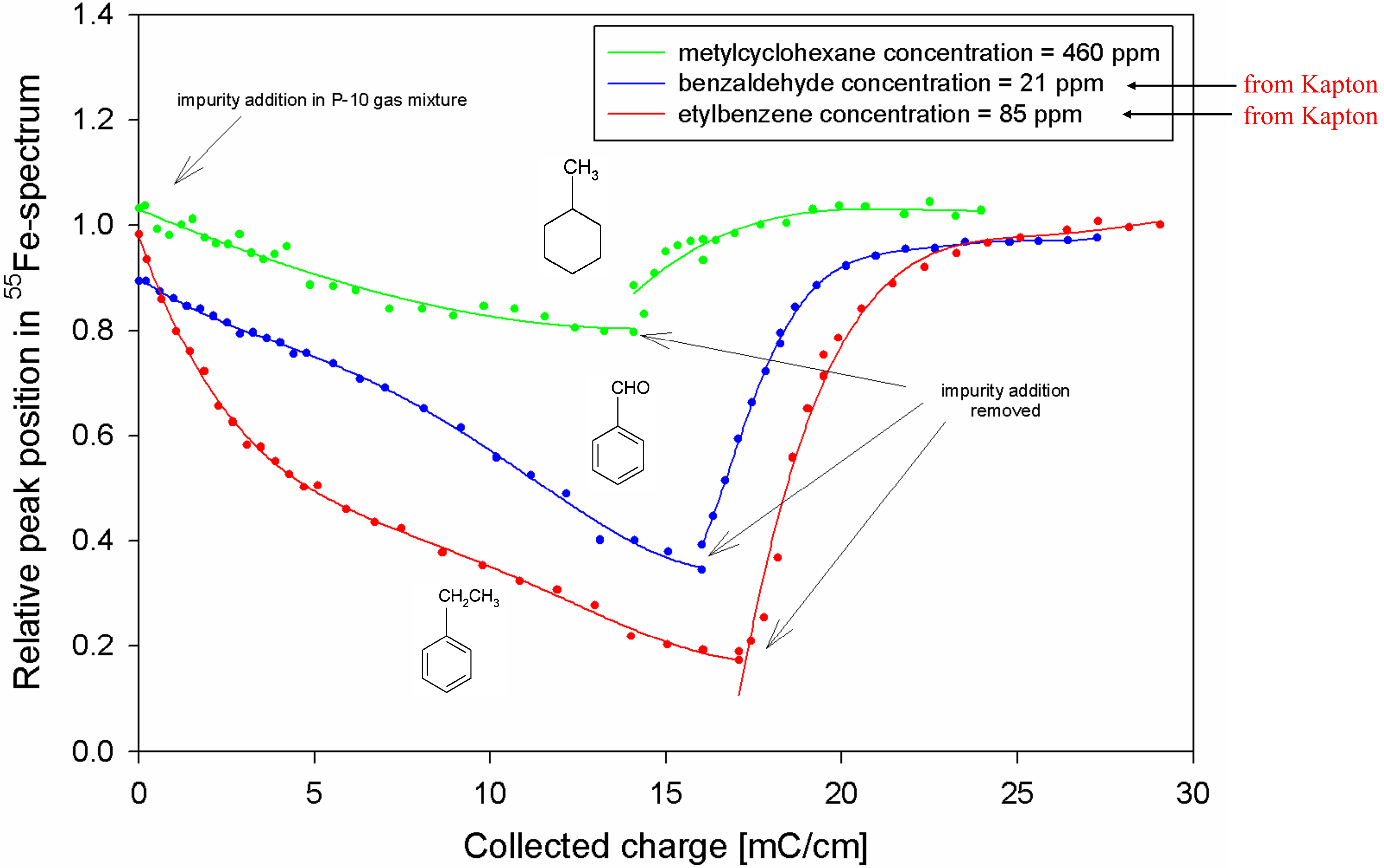


Aging of a proportional counter
with two different impurities of cyclohexane in P-10 gas mixture

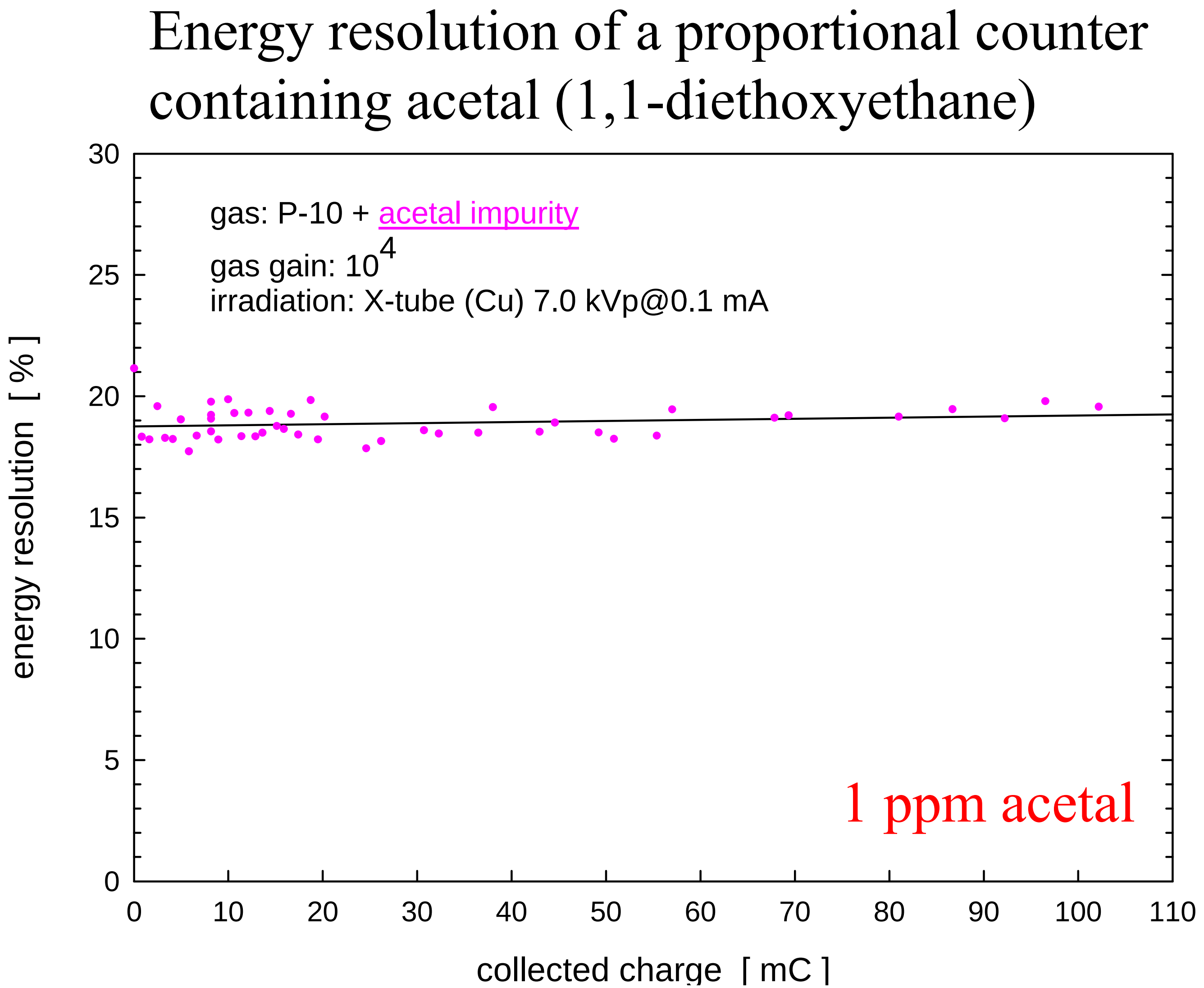


Results - Accelerated Aging Test

Aging of a proportional counters with impurities of metylcyclohexane, benzaldehyde and ethylbenzene in P-10 gas mixture

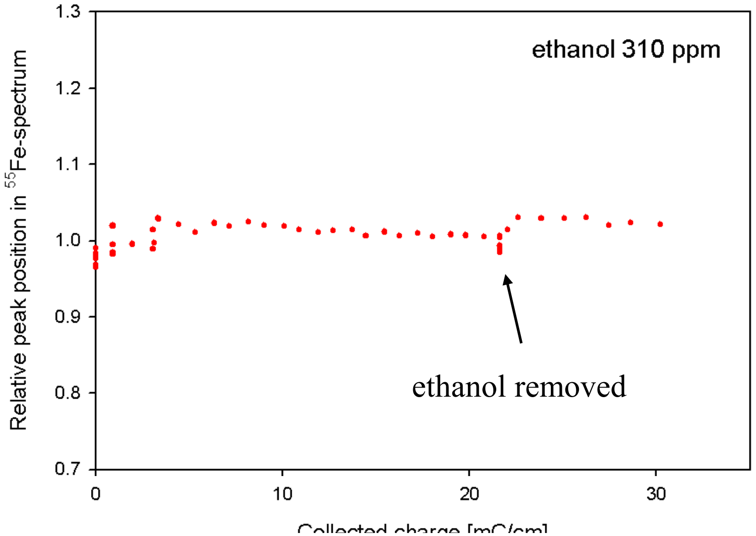


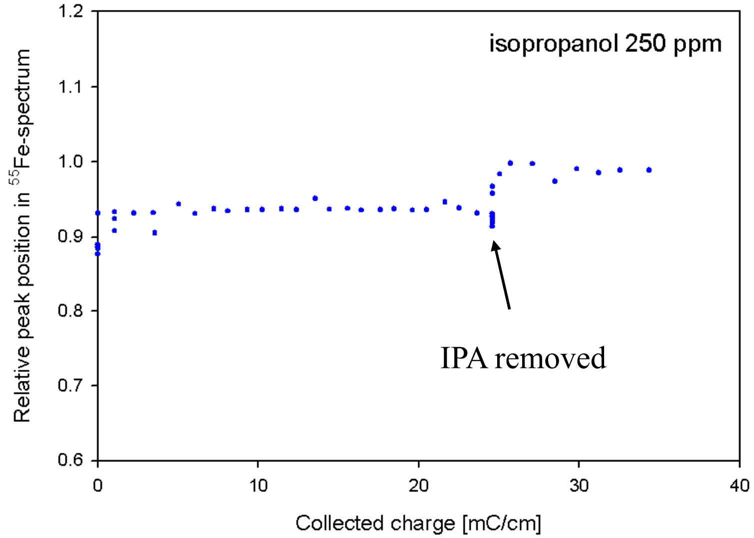
Results - Accelerated Aging Test

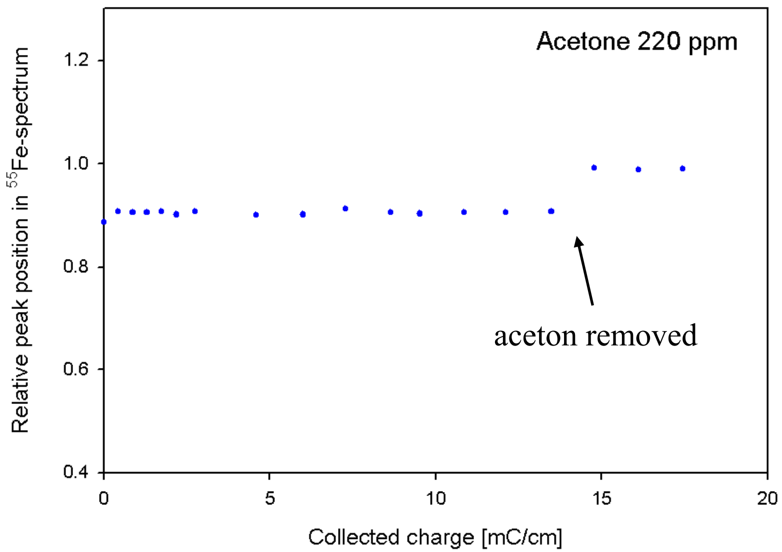


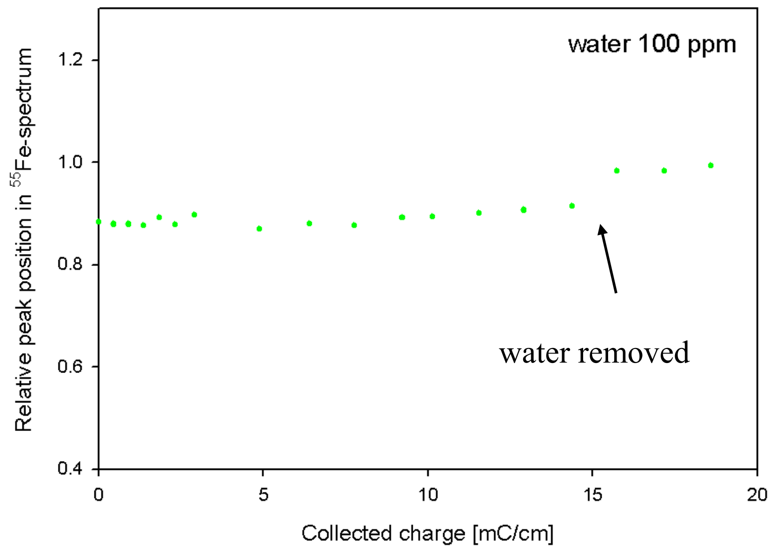
Results - Accelerated Aging Test

Some non-aromatic common solvents









Results – Starting point for a database?

Outgassing compounds from various materials

OUTGASSING YHDISTEITÄ ERI MATERIAALEISTA

yhdiste \ polyimidi	CAS	Kapton HN	Imidex	Espanex	Apical	Mylar	PEEK	Araldit Hardener	Habia cable	RaychenSpec55	FR4	Rilsan
methylenechlorid	75-09-2	X			X							
toluene	108-88-3	X	X	X		X	X	X	X	X		X
tetrachloroethylene	127-18-4	X	X									
xylene	1330-20-7	X	X	X		X		X				X
styrene	100-42-5	X	X									
benzaldehyde	100-52-7	X										
1,2,4-trimethylbenzene	95-63-6	X										
aceticacid-methylester	-			X								
ethylacetate	141-78-6			X	X				X			
N,N-dimethylacetamide	127-19-5			X								
trichloroethylene	79-01-6				X							
2-ethoxyethanol	110-80-5				X							
N,N-dimethylformamide	68-12-2				X							
2-methyl-1,3-dioxolane	497-26-7					X						
1,3-dioxane	505-22-6					X						
1,4-dioxane	123-91-1					X						
1-pentanol	71-41-0					X						
benzene	71-43-2						X		X			
cyclopentanone	120-92-3						X					
N-methylmethanamine	42751-79-1							X				
N,N-dimethyl-2-propanamine	996-35-0							X				
N,N,N-trimethyl-1,3-propandiamine	-							X				
1,1-diethoxyethane	105-57-7								X			
(2-pentanone)	-								X			
(2-buteonicacid methylester)	-								X			
3-hexen-2-one	-									X		
C5H4F8	-									X		
(C8H8F9)	-									X		
(C10H14F8)	-									X		
(C11H20O2)	-									X		
(5,7-dimethylundecane)	-									X		
(2,2,4,4-tetramethyl-3-pentanone)	-									X		
methylbromide	74-83-9										X	
probenebromide	-										X	
2-methylpyridine	109-06-8										X	
3-methylpyridine	108-99-6										X	
2,6-dimethylpyrazine	108-50-9										X	
dimethylbenzamine	-										X	
2-methylbenzofuran	4265-25-2										X	
2-bromophenol	95-56-7										X	
phenol	108-95-2										X	
C6H12O2Si	-											X
C8H22O3Si2	-											X
4-methyl-3-penten-2-one	141-79-7											X
5-methyl-2-hexanone	110-12-3											X

Summary

- Aromatic compounds are observed to outgas from several detector materials.
- All the tested aromatic compounds (benzene, xylene, toluene, styrene, benzaldehyde and ethylbenzene) caused aging in a proportional counter filled with P-10 gas mixture.
- The original characteristics of the detector are recovered by irradiation after removing the impurities (except with styrene)
- No aging was observed with five non-aromatic solvents (cyclohexane, ethanol, isopropanol, aceton and water).