Materials and gases: Lessons for detectors and gas systems

Mar Capeans CERN

Invited review talk at the International Workshop on Aging Phenomena in Gaseous Detectors

nternational Workshop on Aging Phenomena in Gaseous Detectors October 2-5, 2001 at DESY (Hamburg

Outline

- Ageing
- Outgassing studies
- Detector Assembly Materials
- Detector Assembly Procedures
- Components for Gas Systems
- Influence of Some Gases on Ageing



- Gaseous detectors operated in a high radiation environment can have serious operational problems due to ageing.
- Commonly ageing manifests as:
 - Loss of gas gain
 - Worsening of energy resolution
 - Excessive currents
 - Self-sustained discharges
 - Sparking

All these are usually related to the **presence of deposits** coating the electrodes (anode and/or cathode).

Studies Materials Procedures Gas systems

Gases

Basic Knowledge

- The ageing process is due to very complex phenomena that take place during the gas avalanche (~ plasma chemistry).
- These phenomena lead to the creation of a wide variety of species that may accumulate as deposits, conductive or insulating, on the electrode surfaces.



Polymers

- Solid, highly branched and cross linked
- Excellent adhesion to surfaces
- Resistant to most chemicals
- Insoluble in most solvents

Ageing

Basic Knowledge

- Measured ageing, quantitative and qualitatively, depends on many factors:
 - Gas gain
 - Gas flow & Pressure
 - Charge density
 - Geometry and configuration of electric field
 - <u>Active gas</u>

Nature of gasTraces of contaminants

• Experimental observations are difficult to compare and/or extrapolate between one another.

Recent experience

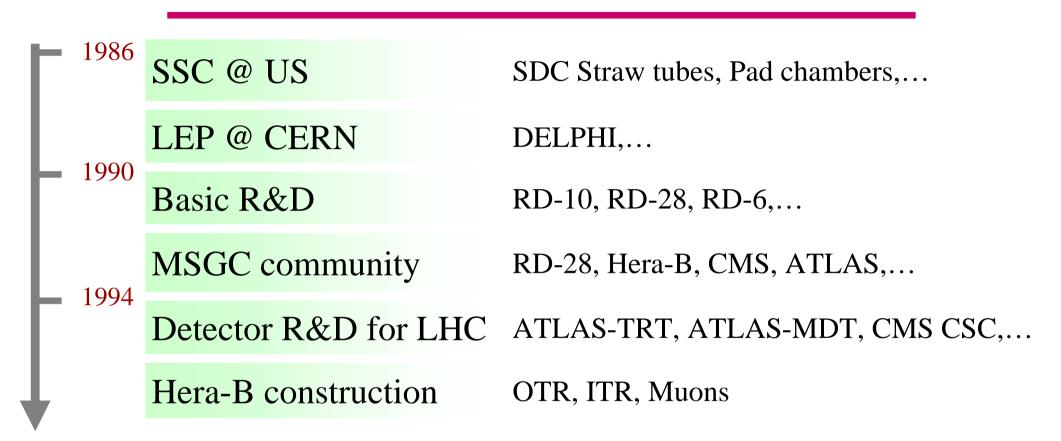
Materials

Studies

Procedures

Gas systems

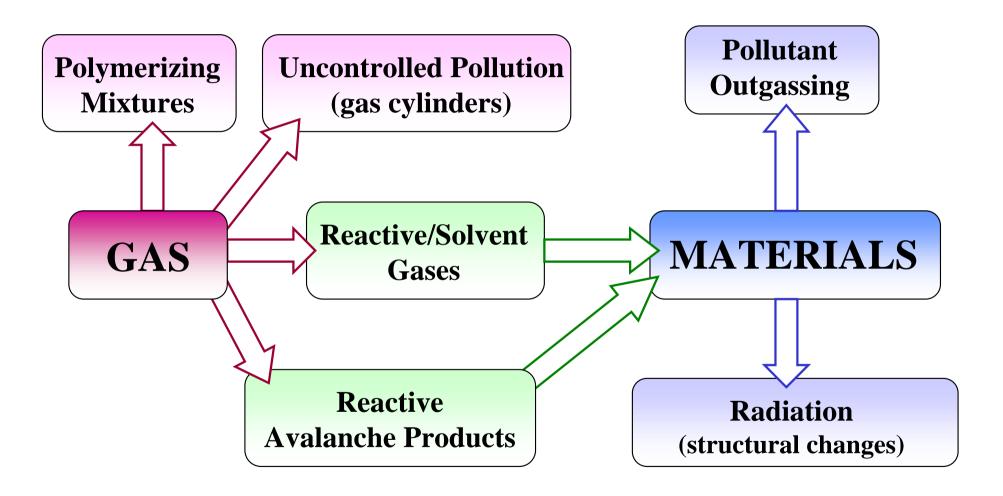
Gases



Still comparison and/or extrapolation is difficult but all this experience provides a solid basis for discussion

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Gases



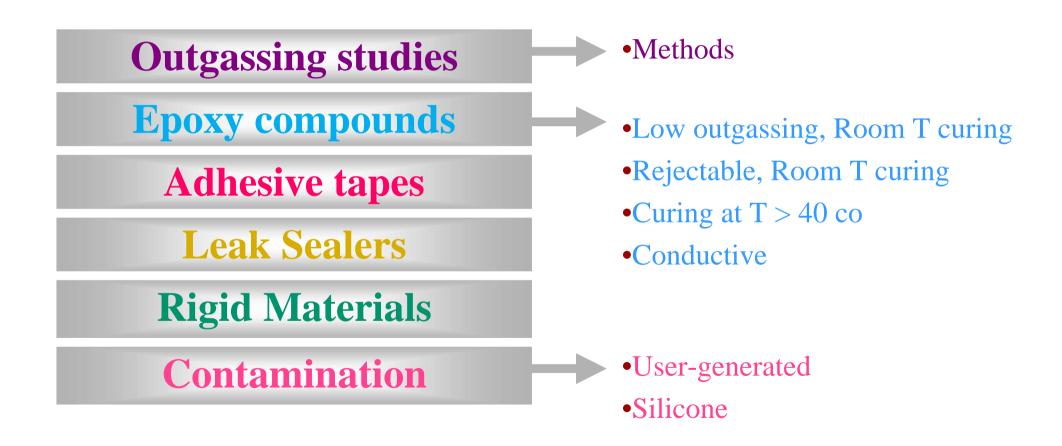
Procedures C

Gas systems

Gases

Outgassing Tests of Some Materials

Materials



Ageing

Studies

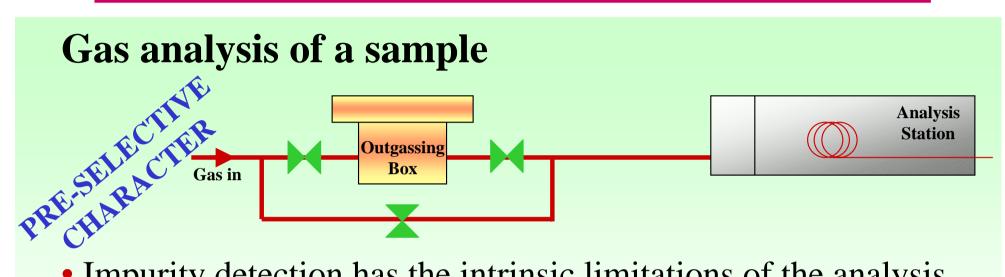
The Minimal Set-Up

Materials

Procedures

Gas systems

Gases



- Impurity detection has the intrinsic limitations of the analysis station, that may not be sensitive to all species because of:
 - low pollutant concentration
 - specific sensitivity

Studies

- Sometimes heating of the sample is needed to detect outgassing (scaling factor unknown).
- Even if outgassing is detected, it might not be harmful for the gaseous detector.

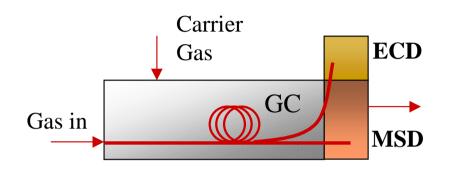
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Ageing

Gas Chromatography Basics

Materials

Procedures



Studies

MSD: 70 eV e⁻ source to ionize and fragment molecules + quadrupole mass filter to sort resulting ions according to their M/Z ratio. Function: it **identifies** each molecular compound. Signal: ion abundance as function of retention time in the column or M/Z ratio.

<u>Sensitivity:</u> ~ ppm

GC: oven with a capillary column. <u>Function:</u> it **separates** gas substances depending on their interaction properties with the column. <u>Signal:</u> a signal appears for each separated compound at some retention time, defined by the column and temperature profile.

Gas systems

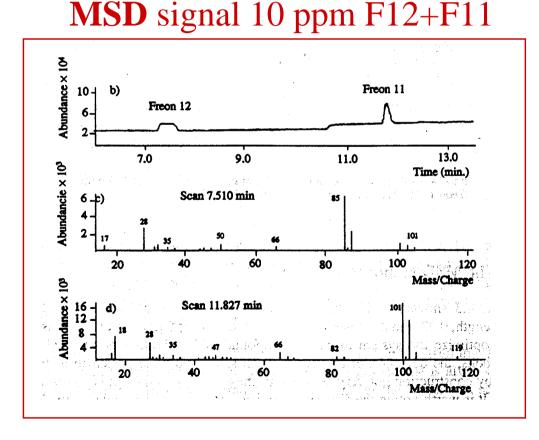
Gases

ECD: βe⁻ bombard a carrier gas to form a plasma. Thermal e⁻ are collected applying an E-field. The decrease of the detector current due to removal of thermal e⁻ by recombination in presence of electro-capturing compounds gives a signal. <u>Function: detection of electronegative</u> substances <u>Signal:</u> signal amplitude vs. time. <u>Sensitivity: ~ ppb</u> **Gas Chromatography Basics**

Materials

Procedures

Selection of column, temperature profile and carrier gas nature and flow defines the sensitivity of the station.

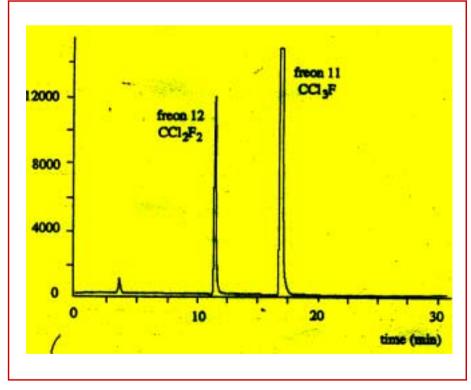


Studies

ECD signal 10 ppm F12+F11

Gas systems

Gases



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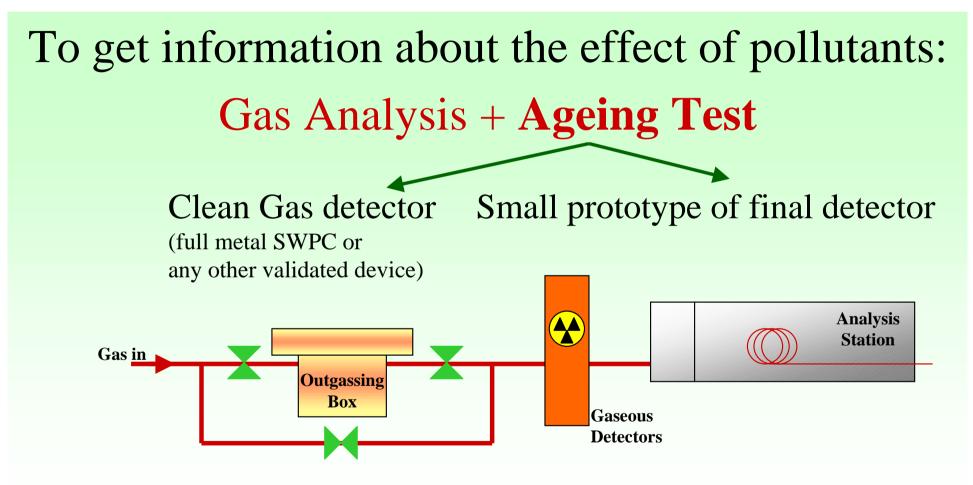
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Procedures

Gas systems

Gases

Materials



Limitations: - Irradiation conditions (high dose rates) - Time scale (~months)

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Ageing

Studies

Studies Materials

TML & CVCM Method

PRE-SELECTICRE CHARACTER **Developed at NASA to search low outgassing materials for** spacecraft use.

http://epims.gsfc.nasa.gov/og/index.cgi

- Test of conditioned micro-quantities in vacuum at stated temperatures and specific times.
- **Technique** of condensing the volatile products to determine the amount of volatile condensable materials.
- Output: Low Outgassing Material TLM < 1% & CVCM < 0.1%

% TML = Total Mass Loss	% CVCM = Collected Volatile Condensable Materials
It is determined from the weights before and after the temperature exposure.	It is the difference between the weight of a clean collector and of the collector having condensed materials.

Epoxy Compounds

Parameters to take into account during selection:

- Shear strength
- Elongation at break

Studies

• Viscosity

Ageing

- Electrical properties
- Capillary effect
- Radiation resistance
- Outgassing

Material itself

- •User-generated (hard to trace):
 - Pollution
 - Incorrect ratio of hardener to resin
 - Insufficient mixing
 - Insufficient curing time

Studies

Low Outgassing Epoxy Compounds Room-T Curing

Source	Product	Outgas	Effect in G.D.	Note
CERN/GDD	STYCAST 1266 (A+B)	NO	NO	Long curing time
HERA-B/OTR	STYCAST 1266 (A+Catalyst 9)	NO	NO	In Use
CERN/GDD	HEXCEL EPO 93L	NO	NO	Out of production
HERA-B/ITR	ECCOBOND 285	NO	NO	In Use
CERN/GDD ATLAS/TRT	ARALDITE AW103 (Hardener HY 991)	NO	NO	In Use
ATLAS/TRT	TRABOND 2115	NO	NO	In Use

Ageing

'Rejectable' Epoxy Compounds Room-T Curing

Source	Product	Outgas	Effect in G.D.	Result
CERN/GDD	ARALDITE AW 106	YES		BAD
ATLAS/TRT	(Hardener HV 935 U)			
CERN/GDD	DURALCO 4525	YES	YES	BAD
CERN/GDD	DURALCO 4461	YES	YES	BAD
CERN/GDD	HEXCEL A40	YES	-	BAD
CERN/GDD	TECHNICOLL 8862 + (Hardener 8263)	YES	-	BAD
CERN/GDD	NORLAND NEA 155	YES	-	BAD
CERN/GDD	EPOTEK E905	YES	-	BAD
CERN/GDD	NORLAND NEA 123 (UV)	YES	-	BAD

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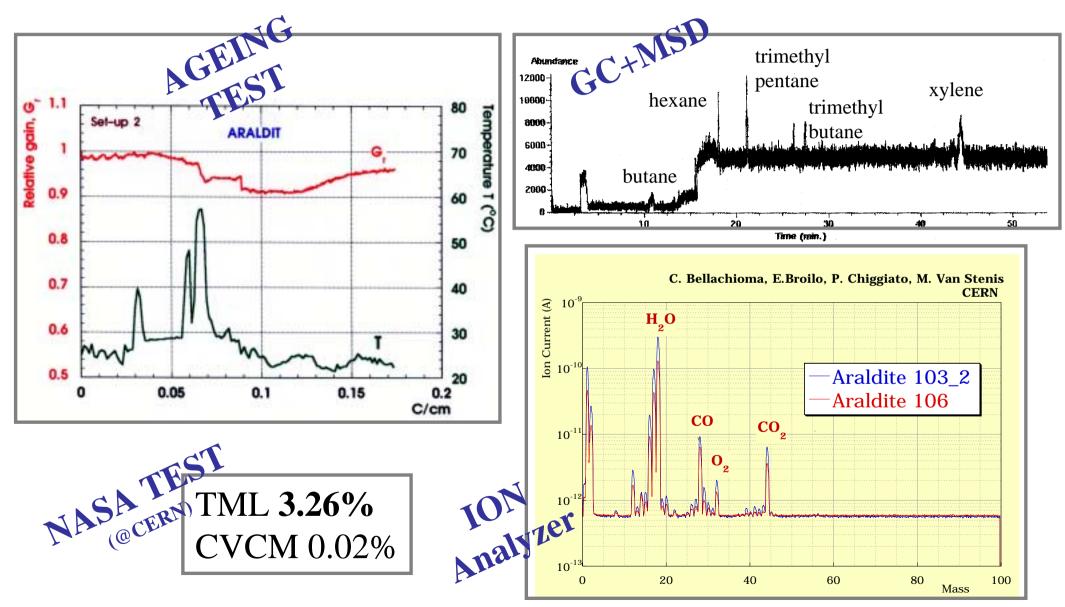
TESTS OF ARALDITE AW 106 (+HV953U)

Materials

Procedures

Gas systems

Gases



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Ageing

Studies

Epoxy Compounds Curing at T > 50 °C

Source	Product	Curing T (°C)	Outgas	Effect in G.D.	Result
CERN/GDD	EPOTECNY E505 SIT	50	YES	NO	OK
HERA-B/ITR	EPOTEK H72	65	YES*	NO	OK*
CERN/GDD	AMICON 125	85	NO	-	OK
CERN/GDD	POLYIMIDE DUPONT 2545	65	NO	-	ОК
ATLAS/TRT	RUTAPOX L20	60	NO	-	OK
CERN/GDD	ARALDITE AW 106	70	YES		BAD
CERN/GDD	LOCTITE 330		YES	YES	BAD
CERN/GDD	EPOTECNY 503	65	YES (Silicone)		BAD
CERN/GDD	NORLAND UVS 91	50	YES	-	BAD

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Stu

Ageing

Conductive Epoxy Compounds

Source	Name	Outgas	Effect in G.D.	Result
CERN/GDD	TRADUCT 2922	NO		OK
HERA-B/OTR	SILBER LEITKLEBER 3025 (A+B)	NO	NO	OK
ATLAS/TRT	TRABOND 2902	NO	NO	OK

Adhesive Tapes

Source	Name	Outgas	Effect in G.D.	Result
HERA-B/OTR	SCOTCH 467 MP	YES	-	BAD
HERA-B/OTR	TESAFIX 4388	YES	-	BAD

Outgassing Tests of Leak Sealers

Source	Material	Туре	Outgas	Effect in G.D	Global Result
CERN/GDD	VARIAN Torr-Seal	Solvent-free epoxy resin	NO	NO	OK
CERN/GDD	RHODORSIL CAF4	Caoutchouc Silicone RTV	NO	NO in very small quantities	OK ?
CERN/GDD	DOW CORNING R4-3117 RTV	Silicone based	YES	NO in very small quantities	OK ?
HERA-B /OTR	LOCTITE 5220	Polyurethane- based	YES	_	BAD

Rigid Materials

Source	Name	Туре	Outgas	Effect in G.D.	Result
CERN/GDD	STESALIT 4411W	Fiberglass	YES	NO	OK
CERN/GDD	VECTRA 150	Liquid Crystal Polymer	YES	NO	OK
CERN/GDD	PEEK Crystalline	Polyeteherether ketone	NO	NO	OK
ATLAS/TRT	ULTEM	Polyetherimide	NO	-	OK
ATLAS/TRT	C-Fiber	C-fiber	NO	-	OK
ATLAS/TRT	POLYCARBONATE	C-fiber	NO	-	OK
HERA-B/ITR	FIBROLUX G10	Fiberglass	YES	-	BAD
HERA-B/ITR	HGW 2372 EP-GF	Fiberglass	YES	YES	BAD
CERN/GDD	RYTON	Polysulphur phenylene	YES	YES	BAD
CERN/GDD	PEEK Amorphous	Polyetherether ketone	YES	-	BAD

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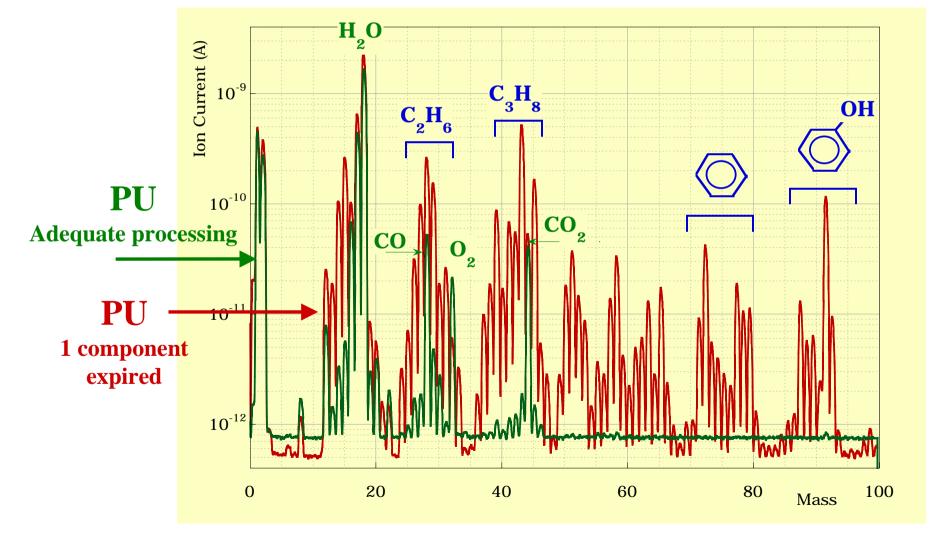
User-generated Outgassing

Procedures

Gas systems

Materials

2-component Polyurethane (Nuvovern LW)



Gases

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Ageing

Studies

AgeingStudiesMaterialsProceduresGas systemsGases

Comparison CVCM, Chromatography, Ageing Test

SAMPLE	NASA	GC	Ageing test
Stycast 1266	BAD	OK	OK
Araldite 103	BAD	OK	OK
Araldite 106	BAD	BAD	BAD
Eccobond 285	ОК	OK	ОК
Nuvovern LW PUR	ОК	OK	ОК
ULTEM	ОК	OK	ОК
VECTRA 150	ОК	OK	ОК
Kalrez	ОК	OK	ОК
Epotek 905	BAD	BAD	
Dow Corning RTV	BAD	BAD	

Consult NASA database to select materials, before doing the time-consuming tests NASA Database: More than 1600 entries for adhesives, 500 entries for rubbers and elastomers, 800 entries for potting compounds, etc...

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Silicone Contamination

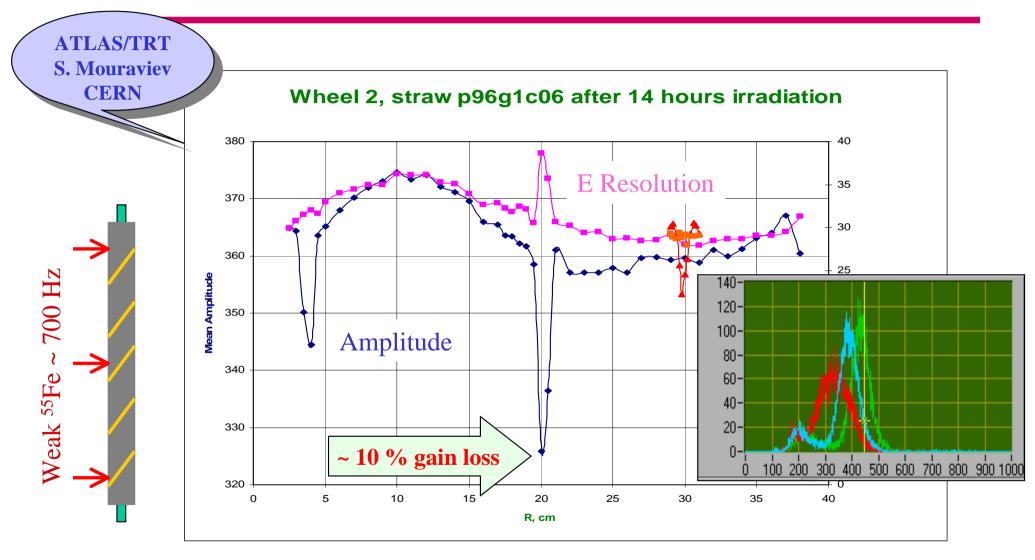
- Silicone has been systematically found coating aged chambers.
- Silicone has a high natural affinity for most materials.
- Silicone has the tendency to migrate.
- Silicone is relatively inert chemically and unaffected by most solvents, therefore among the most difficult surface contaminants to remove.
- Silicone is etched away by F-species.

Possible Sources:

- Silicone rubber sealants
- Silicone potting and encapsulation compounds
- Silicone adhesives
- Silicone Vacuum Grease (O-rings, mould-release agents)
- Silicone oil (bubblers, diffusion pumps)
- Polluted gas cylinders
- Detergent residues (sodium metasilicate)
- Glass and related products (glass fibres used for reinforcing resins)

Ageing

Accidental Silicone Contamination



Ageing

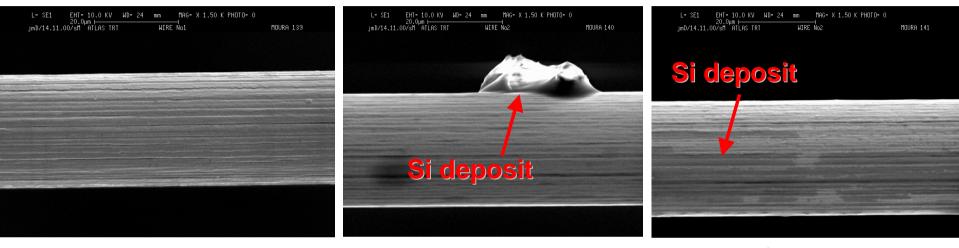
Materials

Procedures

Gas systems

Gases

Silicone Contamination



Clean wire

Irradiated spot

Region between two irradiated spots

Silicone Source:

Silicone-based lubricant used accidentally during straw tube manufacturing by the manufacturer.

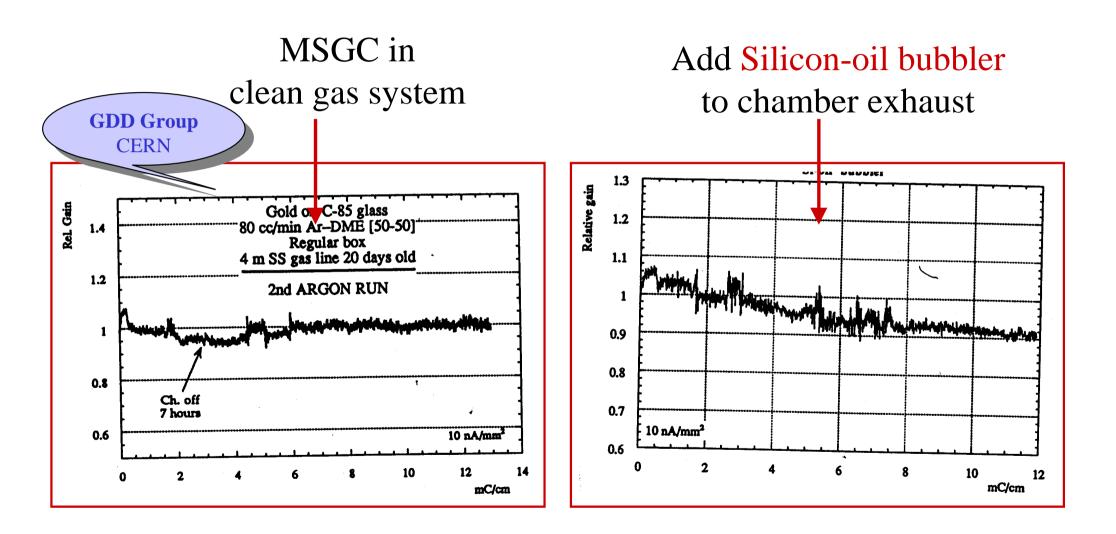
Mass production straws are free of pollution (no lubricant at all).

Materials

Procedures

Gas systems

Gases



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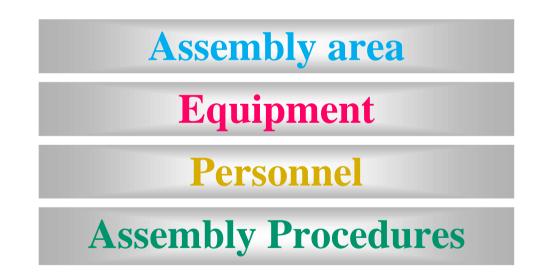
Ageing

Studies

AgeingStudiesMaterialsProceduresGas systemsGases

Detector Assembly Procedures

Contaminants during the assembly process such as dust particles or an invisible film of organic material can make the best-designed detector fail.



Assembly Area

Procedures

Materials

Studies

The assembly area must be **isolated** from other manufacturing areas, and usually following clean room WHICH standards. LEVEL

4 assembly sites located worldwide:

Nb. of particles $> 0.3 \mu m$ in a 0.1 cubic foot of air

Office: 440000 Std hall: 770000 Area 1: 1 - 20 Area 2: 1 - 15 Area 3: 760000 Area 4: 520000

Gas systems

Gases

All modules behaved equally well during HV testing and running in the Hera-B environment.

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Gas: Ar-CO₂-CF₄

Detector: drift tubes of small diameter

100 krad/y

2 dose

 $3x10^5$ Hz/cm²,

2 Rate

Ageing

Hera-B/OTR

Desy

Ageing Studies Materials Procedures Gas systems Gases

Equipment

Some equipment should be avoided or protected

Examples of contamination are:

• Tools with lubricated shafts, cranes...



- **Soldering** or brazing equipment that require heating of volatile fluxes
- Motors and vacuum pumps with outgassing oils
- Some electronic equipment contain capacitors, resistors, etc coated with organic or insulating materials that outgas

Personnel

Procedures

Usual source of contamination.

Need careful and exhaustive **training**.

Examples of contamination are:

Studies

• Street clothing (source of lint)



Gases

Gas systems

• Hair, make-up, fingernails (source of oil and particulates)

Materials

- Many hand creams and cosmetics (contain silicones)
- Saliva
- **Fingerprints** (source of fat, possible cause of corrosion in some metals, etc)

Assembly process

It has to:

- Be well specified and stable
- Be rechecked periodically
- Include specification for the storage of parts
- Include procedures about:
 - cleanliness of assembly components
 - verification of cleanliness
- Include tracing capability
 - operator ID, assembly step, material batch, environmental parameters, etc

Gases

About Gas System Components

Compatibility Aspects

Clean Components

The Golden Rule

- Validation
- P-regulators
- Flowmeters
- Valves
- Connectors
- Piping
- Elastomers

Gas systems used for:

Procedures

Gas systems

Gases

Materials

- Assembly procedures
 - Wiring, etc

Studies

- Cleaning of components (N₂ or compressed air guns)
- Cooling of assembly set-ups
- Polymerization of glues
- Others
- Acceptance tests at the laboratory
- Final gas systems in experimental area

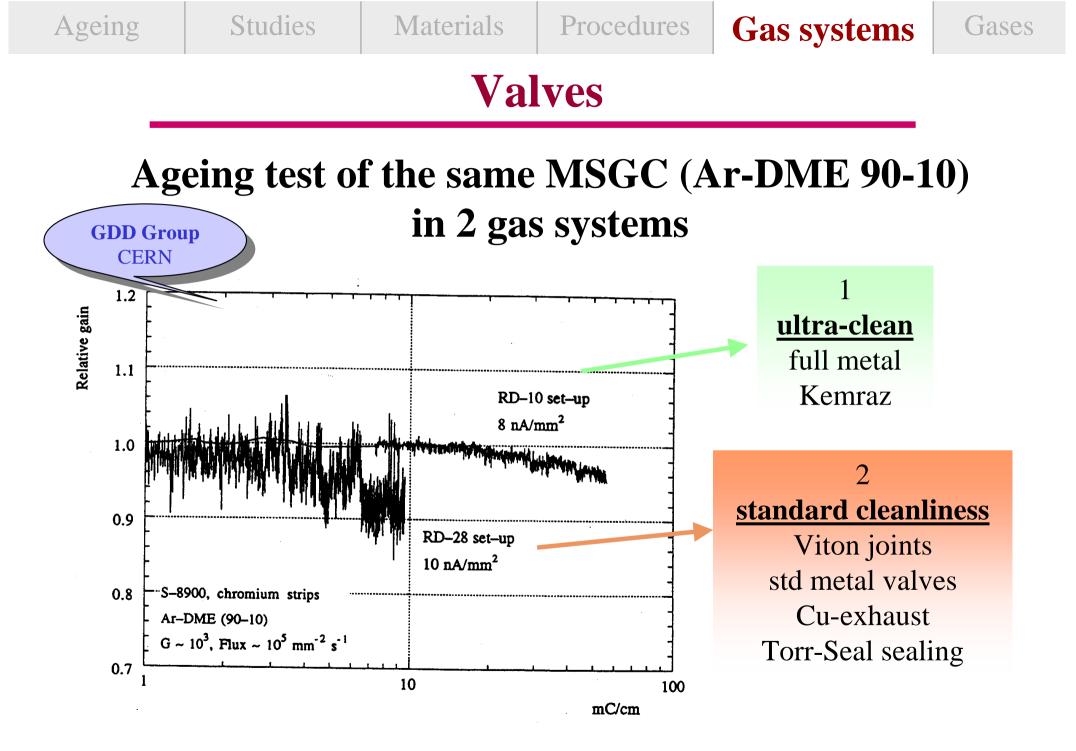
Compatibility

- Materials VS Ageing
- Chemical compatibility with operating gas
- Purity and cleaning of components
- For final gas systems:
 - Radiation

Test of electronic components

• Magnetic field

Avoid electro-magnetic valves, electro motors, relays. Check control valves, pressure sensors, power supplies, etc.

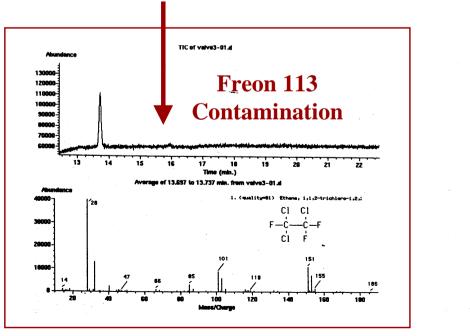


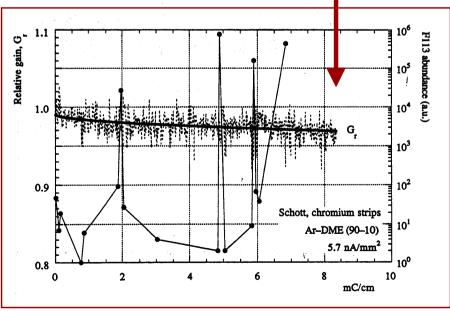
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Valves

Systematic study (GC) to search the origin of pollution

- Isolate components of the std.
 gas system
- Manual SS ball-valve (PFTE joint) found suspicious
- Take valve to ultra-clean setup
- Ageing Test + accelerated rate of outgassing





AgeingStudiesMaterialsProceduresGas systemsGasesSearch for Clean ComponentsThe ATLAS-TRT approach:• Problem:

- straw tubes are very sensitive to silicone pollution
- Approach: use only clean components

Market-survey to look for clean gas components equivalent to ultra-high purity standards in terms of very low outgassing rates and absence of lubricants (especially silicone-based). The requirements have to be discussed with the companies. **Materials**

Components

• Cylinder P-regulators

Studies

• Filters

Ageing

- Flowmeters
- Valves
- Exhaust
- Piping
- Connectors

Validation of Components Straw tube AGEING Tests

Gas systems

Gases

• Nominal gas:

Procedures

- Xe-CF₄-CO₂ 70-20-10
- Time Length: 1000 hours
- Dose:1 mm collimated source ⁵⁵Fe, 5 KHz

Materials

Procedures

Studied in detail 15 different models from ~10 companies.

Knowledge	Price CHF
Ultra-high purity applications (semiconductor industry):	> 2000
Lubricant-free certified & excellent surface quality.	
Reducing our requirements (in terms of surface quality): - Brass option at SCOTT Specialty Gases (model C21-8) -Parker Veriflo line (model IR4002)	~ 600
High purity applications:	>1000
Lubricant-free certified, but experience tell us that it might NOT be true.	
Some companies produce affordable, clean products: TESCOM (serie04)	~ 600
Standard applications: They might be certified lubricant-free by suppliers For safety, they always need additional cleaning	~ 300 - 600

Ageing

Studies

Gases

Gas systems

Pressure Regulators

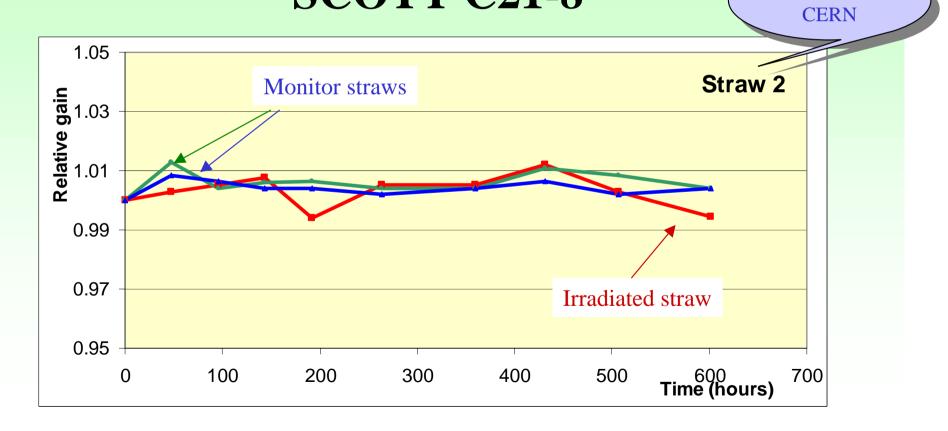
Materials

Procedures

Gas systems

Gases

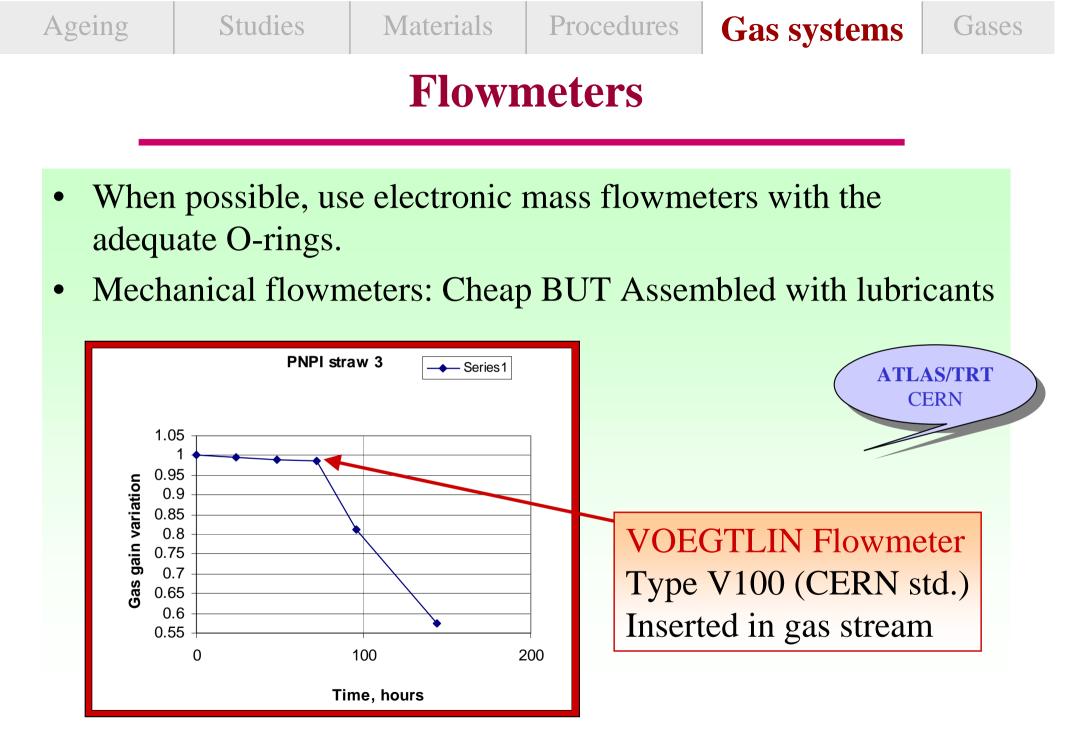
Validation of P-regulator dual stage SCOTT C21-8



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Ageing

Studies



Flowmeters

Procedures

Cleaning flowmeters

Studies

Ultrasonic cleaning of dismounted pieces.

Materials

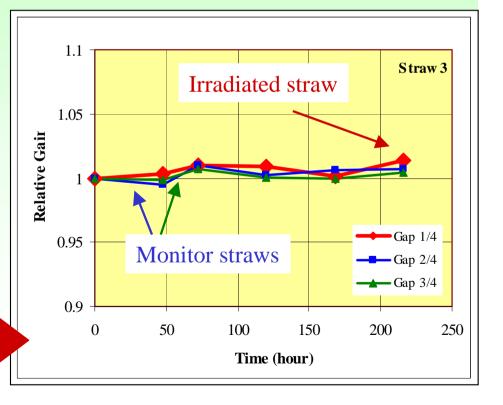
M.Bosteels (CERN):

Ultrasonic bath Isopropyl alcohol 15' Ultrasonic bath Isopropyl alcohol 15' Ultrasonic bath ultra pure water 20 h at 70-80 °C

CLEANED VOEGTLIN Flowm. Type V100 (CERN std.) Inserted in gas stream

EMO

Ageing



Gas systems

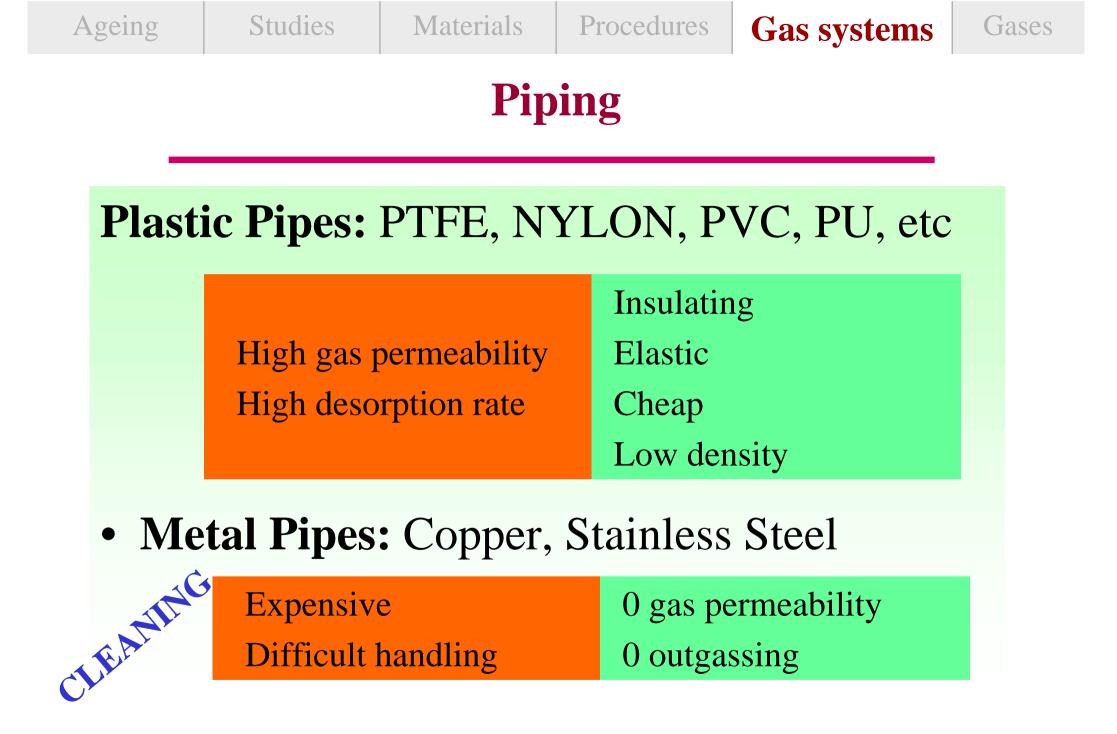


Gases

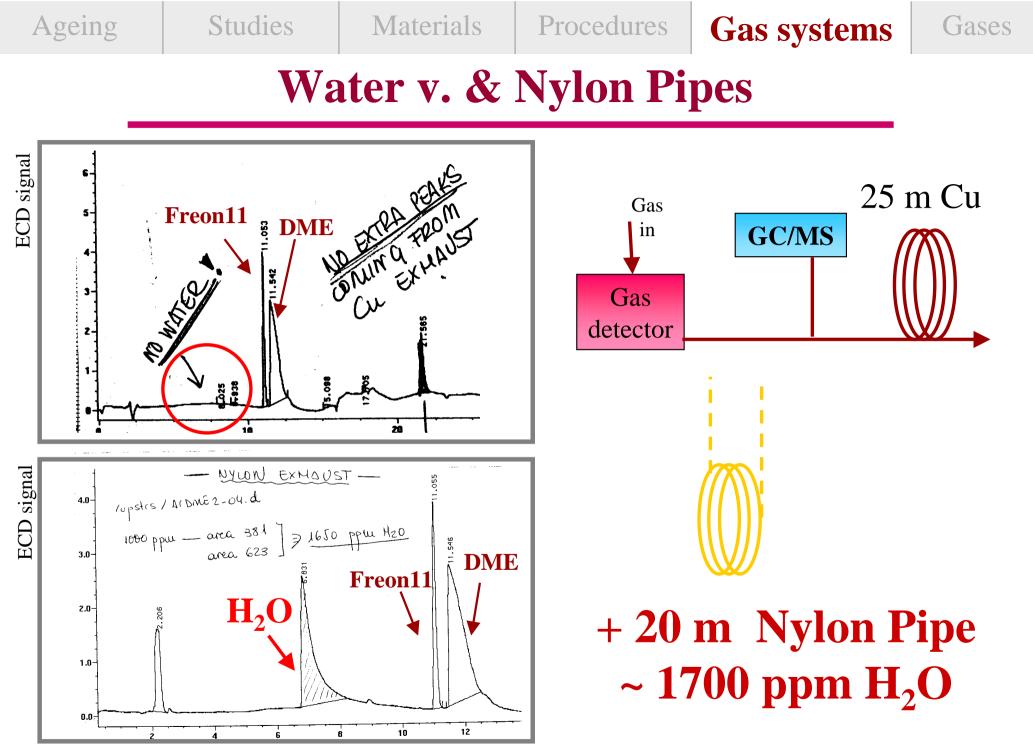
Age	eing	Studies	Materials	Procedures	Gas systems	Gases		
Connectors								
Unions, elbows, nuts, etc								
Company		Note						
SWAGELOK				Certified lubricant-free. If additional SC-11 cleaning, price is 25% higher.				
	GYR	OLOK	Risk of parat	ffin contaminatio	n			
	SAG	ANA		degreased" (= ace	<i>on</i> ' items should be etone baths).	order		

Mount system following clean procedures: protective clothing, clean storage, no open ends, etcFollow manufacturer directions to the letterNever mix one brand or metal with another one

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Agein	g Stud	es	Materials	Procee	dures	Gas syst	ems	Gases
	Outgassing Tests of Plastic Pipes) Group CERN
	Material		Туре	Outgas	Effect in Gaseous Detector		Global Result	
	PP	Poly	propylene	NO		NO	OK	
	RILSAN NYLON	Pol	lyamide	Water		NO	OK*	
	PEEK Crystalline	-	etherether tetone	NO		NO	OK	
	PEEK Amorphous		etherether tetone	YES		-	BAD	
	PEE			YES		-	BAD	
	PUR	Poly	urethane	YES		-	BAD	



Outgassing Tests of Elastomers

Material	Туре	Outgas	Effect in Gaseous Detector	Global Result
KALREZ	Fluoropolymer	NO	NO	ОК
VITON	Fluorinated copolymer	YES	YES	BAD
EPDM	Copolymer ethylene propylene	YES	_	BAD
PVDF	Fluorinated polyvinyldene	YES	-	BAD

Ageing

GDD Group CERN

Gases

Gas systems

About system components...

Procedures

Materials

- It is common to find that manufacturing specifications and product descriptions do not contain enough information.
- Once an adequate product has been found, it is difficult to asses the cleanliness conditions for large quantities.

Ageing

Studies

The Golden Rule

Procedures

Gas systems

Gases

- Define your cleanliness requirements
- Avoid pollution during assembly

Materials

• Flush gas through the open system

AS SOON AS POSSIBLE AS MUCH AS POSSIBLE

Ageing

Studies

Gases

Influence of Some Gases on Ageing

Hydrocarbon mixtures

DME & CO₂

CF₄

Hydrocarbons

Polymerization guaranteed

Procedures

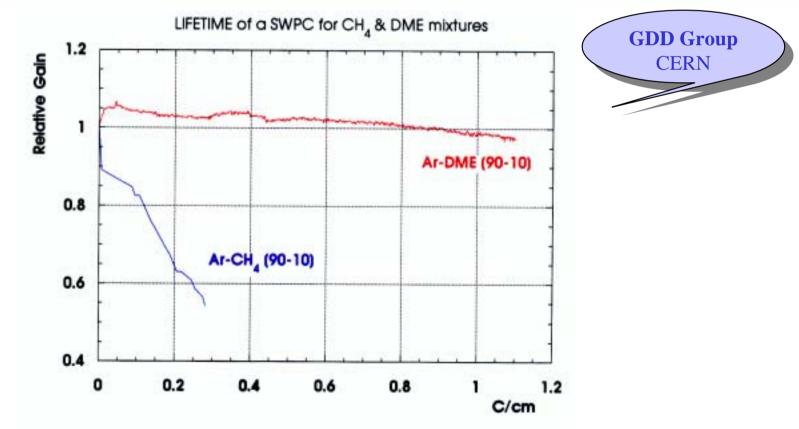
Gas Systems

Gases

- Polymer formation directly in the avalanche process.
- Effect is more pronounced under spark/discharges.

Materials

Studies



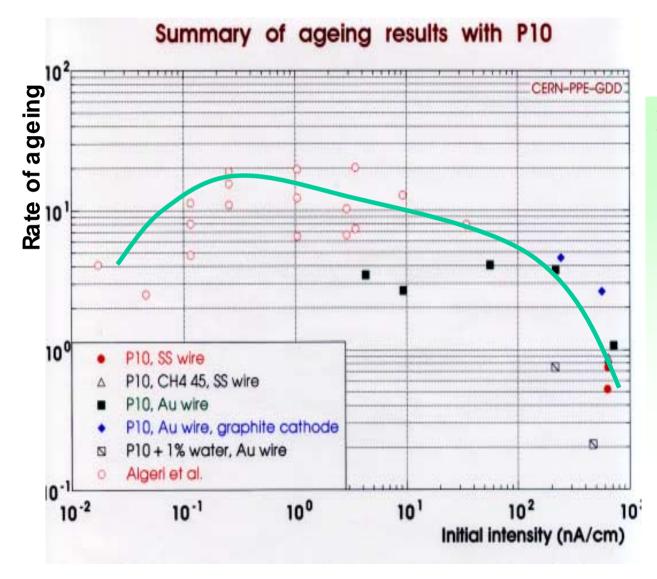
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Ageing

Ageing

Gases

Hydrocarbons



RATE OF AGEING:

- It depends on the charge collection rate.
- ✓ It is independent of electrode material & purity of methane for a given set of irradiation conditions.
- It improves if water (% level) is added.



DME

Advantages

- Small Lorentz Angle
- High primary ionization
- Low Z
- Good quenching properties (safe operation at high gains)
- Wide efficiency plateau
- Rad hard

Disadvantages

- DME is very reactive
- Sensitive to traces of pollutants

Studies

Ageing

Gases

DME

DME is reactive. A careful selection of materials for detector construction and gas system is mandatory.

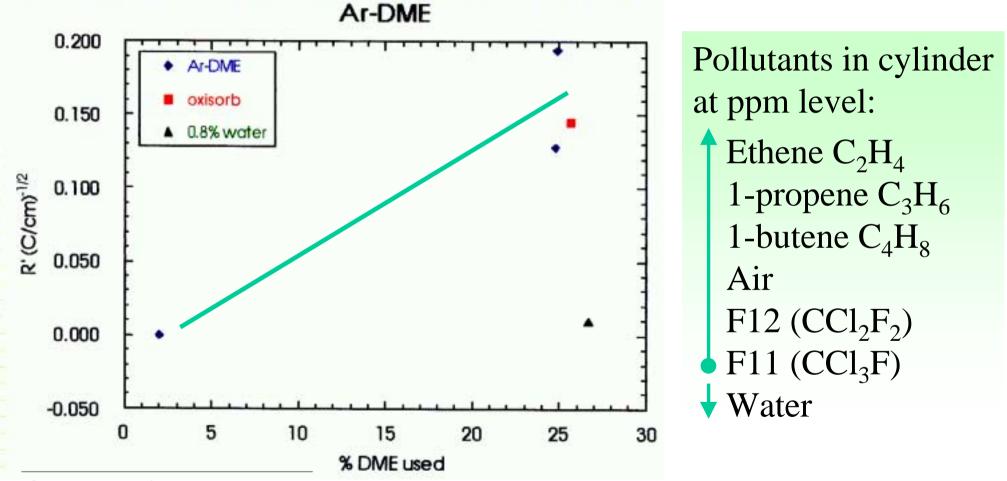
SWELLING (%) in liquid DME for 2 hours

LDPE (foil)	- 0.6			
Vectra	0.0		Ilie ERN	
PEEK Cryst. (pipe)	+ 0.3			
Kalrez (joint)	+ 1.6			
PE	+ 1.8			
Kel-F (joint)	+ 4.0			
PCTFE (joint)	+ 4.0			
PVDF (joint)	+ 5.0			
Kapton (foil)	+ 5.4			
PEEK Amorp. (pipe)	+ 8.7			

AgeingStudiesMaterialsProceduresGas SystemsGases

DME

Ageing rate as a function of DME cylinder consumption



Other Possible Pollutants: Material outgassing (Teflon, PVC, Neoprene, Viton, etc), residuals of cleaning solvents, etc



- High sensitivity to traces of pollutants at ppb level (difficult to control)
- High reactivity with materials (such as Kapton, widely used in Micro-pattern Gas Detectors)

DME has been widely replaced by CO_2 the cost being: Increase of High Voltage Larger energy of discharges



CF₄

Attractive properties

(even for mixtures with a relatively small fraction of CF₄)

- Very high e⁻ drift velocities
- Low diffusion constant
- High primary ionization
- Good ageing properties

Disadvantages

- Etching properties
- Cost (implies gas recirculation for large systems)
- Low E-resolution due to e⁻ attachment

Gases

CF₄: Etching/Deposition balance

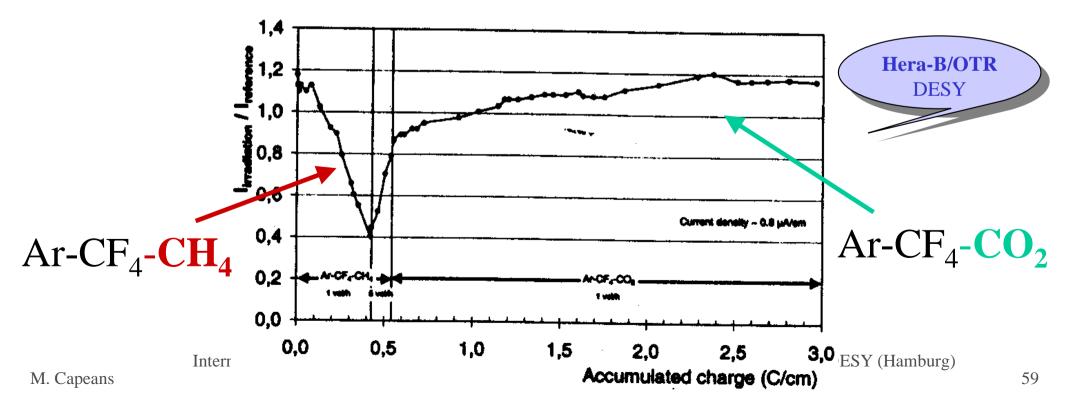
Plasma processing: CF_4 -based gases are used for both etching and deposition processes.

Chemistry is shifted to:

Deposition

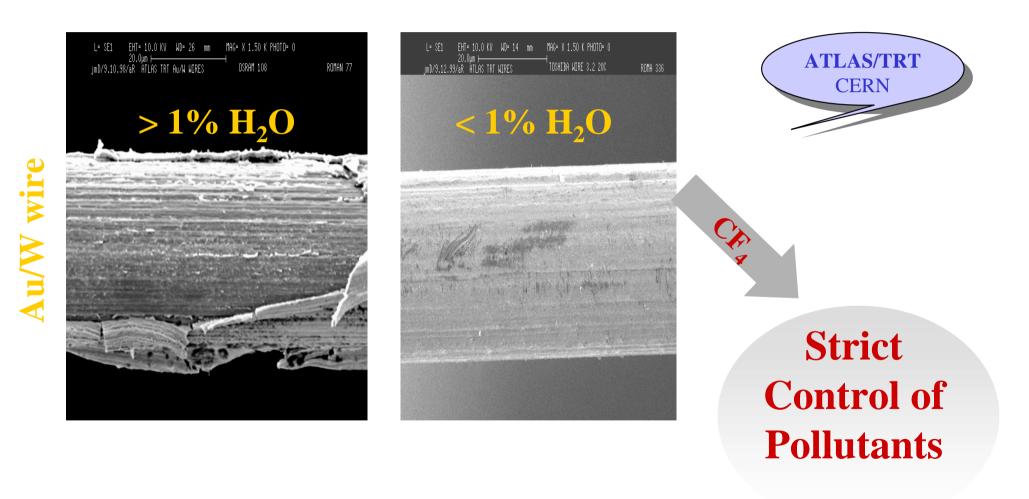
in hydrogenated atmospheres

Etching if oxygenated species are added



Gases

Effect of Water on CF₄-mixtures



Ageing Gas Systems **Studies** Materials **Procedures** Gases **CF**⁴ Etching CF_4 dissociates in F and CF_x radicals (~ stable species). XR Effect of the straw irradiation on the SWPC 400 Straw tube 2 MHz/cm 4mm diam. 350 Xe-CO₂-CF₄ Radiation on 300 50-30-20 250 200 SWPC Pulse height 100 **SWPC** 50 XR ~ 3 cm drift 0 ~Hz/cm 20 0 100 ADC channel

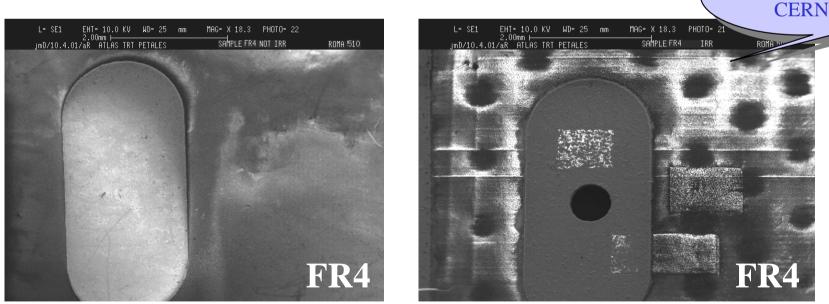
- They have enough energy to break chemical bonds in polymers.
- Polymer is reduced to stable, volatile products.
- These products are removed from electrodes by the gas flow.



CF₄ Etching

Effect on assembly materials:

- Active species react with some metals (Al, Tin) and some insulator materials (Fiberglass).
- F species react with Si, which is distributed all around (polymerization trigger)



After radical exposure

International Workshop on Aging Phenomena in Gaseous Detectors October 2-5, 2001 at DESY (Hamburg)

Ageing

Materials and gases: How to build invincible detectors and gas systems

Extensive R&D is needed, as the experience of other groups is useful but has only a **pre-selective** character.

Detector R&D

• Efficient R&D defines:

- Gas mixture
- **Gas purity**, which implies also careful selection of gas system components (test stations & final system)
- Gas gain
- Assembly materials
- Assembly procedures (avoid user-generated contamination)
- The **long-term capability** of a detector can only be extrapolated to real conditions if final prototypes are:
 - Tested under conditions as close as possible to the final ones, and strictly controlled
 - Large irradiation area & charge density as close as possible to final experiment

Conclusions on Materials

- There is no good or bad material. A **material is adequate** or not for a very particular type of detector and conditions of use.
- Existing data, obtained either from systematic outgassing studies or experience gained with detectors, has only a **pre-selective character**.
 - A list of **low outgassing assembly materials** exists, that includes epoxy compounds, rigid materials, sealants, elastomers, ...
 - The effect of materials that outgas at the ppm level has to be tested for each particular case. That is the case for materials such us Viton, Teflon, Polyurethane, etc
- It seems rather universal that **Silicone compounds** that easily migrate (lubricant-type) should be avoided, especially with some gas mixtures (F species).

Conclusions on Gases

- Noble Gas + **Hydrocarbon**:
 - Not trustable for long-term high rate experiments
 - It is accepted that oxygen containing additives can improve detector lifetime in such mixtures
- Noble Gas + **DME**:
 - They are radiation hard when careful material selection has been done
 - Halogen contamination at ppb level shortens detector lifetime. It is difficult to monitor
- **CF₄**-containing mixtures:
 - They are very attractive in terms of ageing due to the fine polymerizationdeposition balance phenomena
 - They need strict control of pollutants (H_2O)
 - F-radicals are rather stable and able to attack some materials. They are capable of etching away Si-compounds that may trigger strong polymerization processes

