Outgassing and Leak Test studies in INO RPC Detectors

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Outline of Talk

- Leak Test Study
- Aging of gaseous detectors
  - Potential aging effects
- RPC detector at DU
- Aim of the study
- Experimental test facilities
- Gas Analysis
- Results
- Conclusion

Introduction

- INO (India-based Neutrino Observatory) is a proposed underground HEP experiment at Theni, India.

- ICAL@INO 50 kton magnetized detector requires 30,000 2m X 2m RPCs (Resistive Plate Chambers) for 20 years ➔ more than 200,000 litres of gas mixture

- Leakage of the gas mixture increase the cost of operation. Also, the leakage of outside atmosphere into the system will contaminate the gas mixture by water vapour and oxygen, which damages the RPC.

- Minimising wastage of gas which reduces the operational cost

- Due to long-duration of the experiment, each RPC needs to be performed without showing any significant aging during operation.

- Hence, various tests, including a proper leak test are performed.

Leak Test

- ➢ Conventional Manometer only valid when both the volume of the test RPC gas gap and ambient pressure are kept constant during the test period.

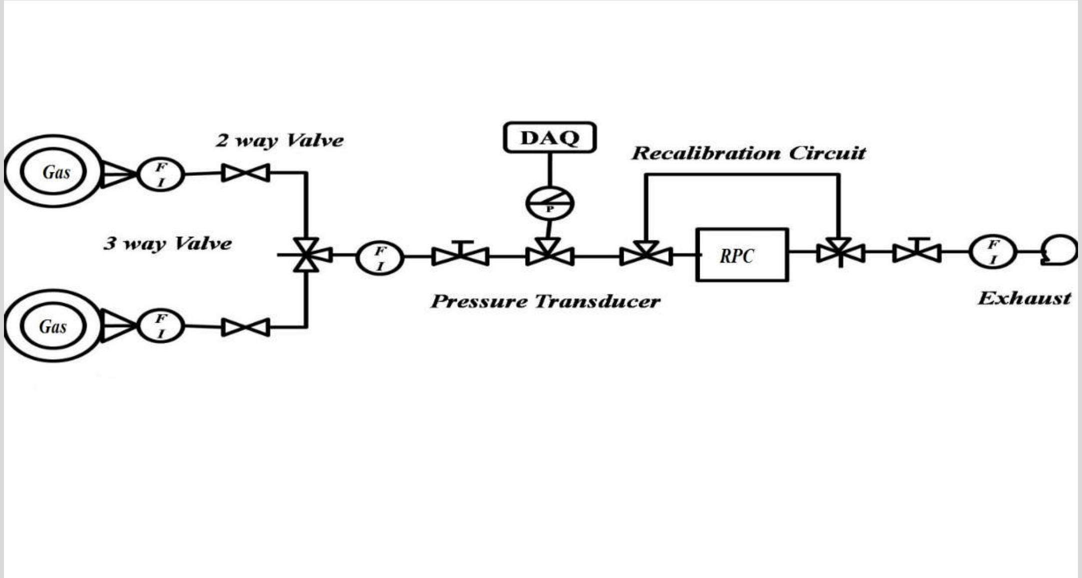
- ➢ A proper quantitative estimation of the leak test estimated by monitoring absolute pressures, both outside and inside of an RPC along with the temperature.

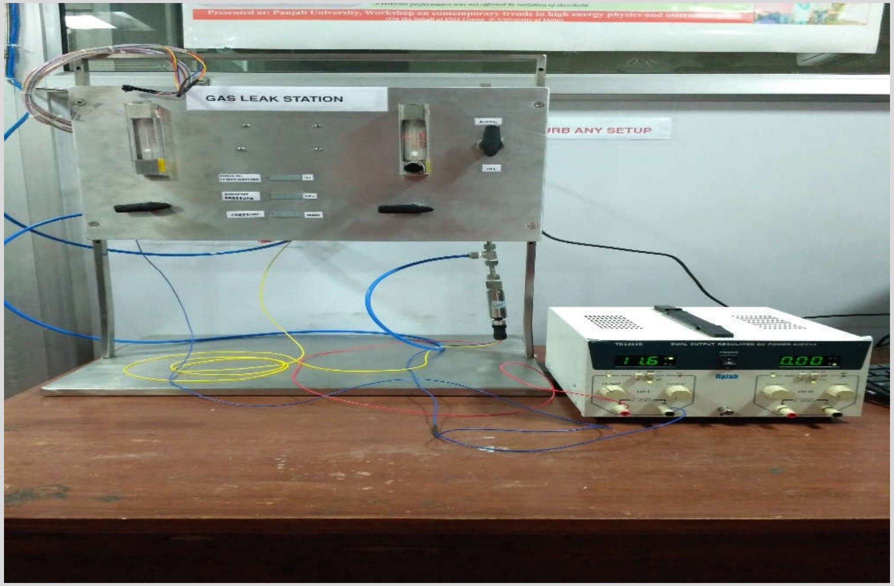
- ➢ Common Method – Pressure Variation (Overpressure method) Measure rate of pressure loss in closed volume.

- ➢ Flux though leak depends on conditions like temperature, pressure difference, gas type.

Leak Test Schematics
& Experimental Setup

- Gas leakage setup uses different electronic and pneumatic components: flow meters, gauge pressure sensor, atmospheric pressure sensor, temperature sensor, digital display, Arduino board, etc.
- Flow meters: measures flow rate of gases
- Gauge pressure sensor (piezoelectric): provides current output for given gauge pressure and measures the corresponding pressure drop
- Atmospheric pressure and temperature: piezoresistive, monolithic, signal conditioned silicon sensor
- Arduino Mega 2560 board: open source computing platform interfaced to PC. The pressure and temperatre data readout by ARDUINO MEGA 2560





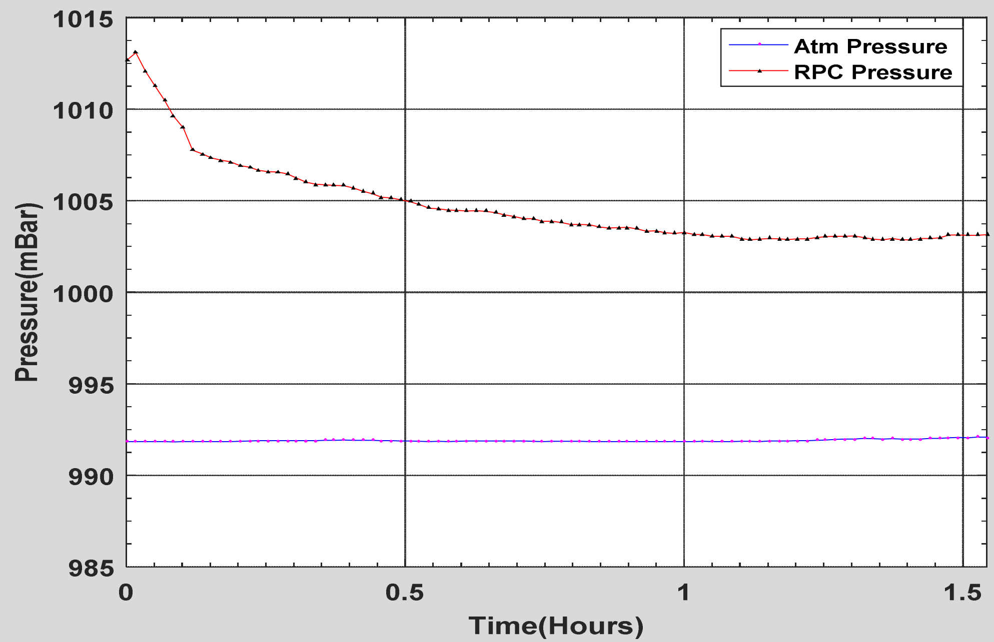
Leak Rate Calculation

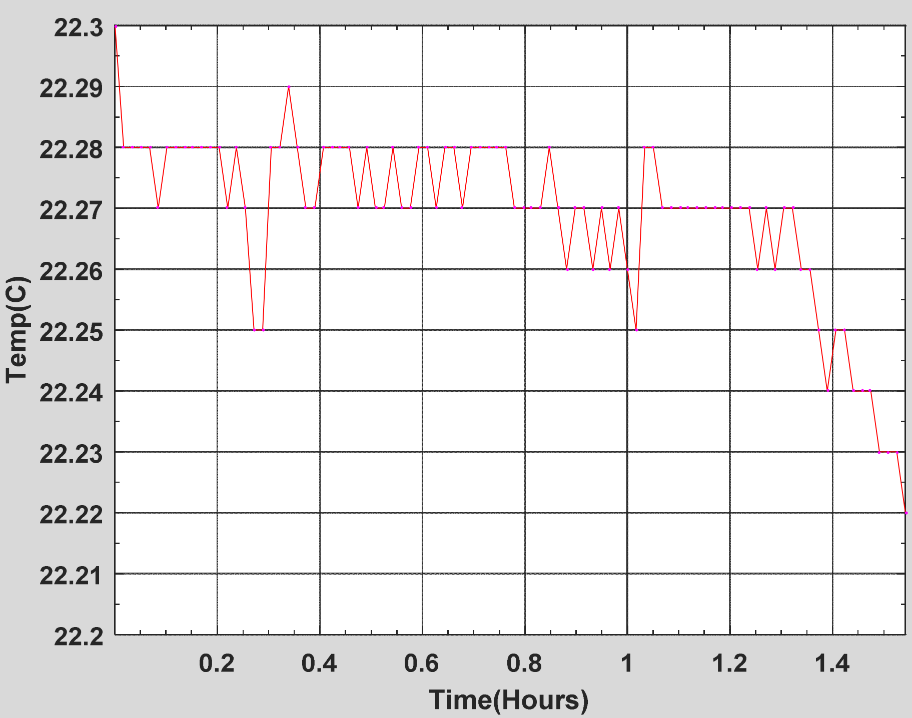
- Gas Ideal Gas Law $n = PV/R\mathrm{T}$

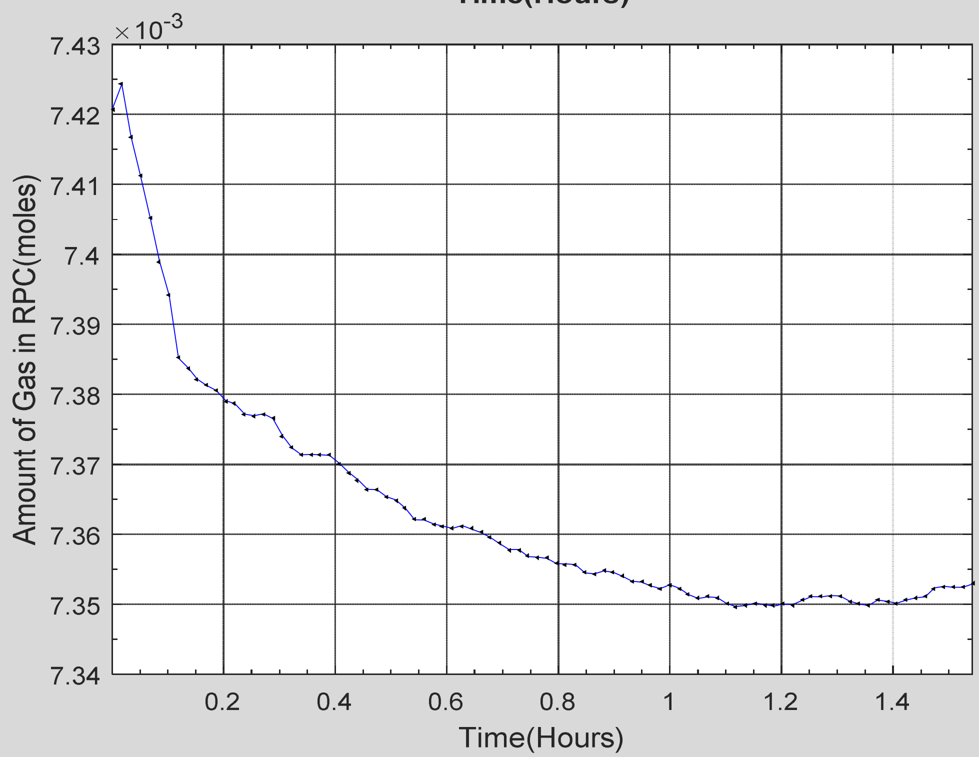
(Ideal gas constant= 8.314 J/(mole.K)

Volume of RPC 0.18 litres

- Pressure inside the gap follows the trend of atmospheric pressure.
- This implies that the volume of the gap changes with the change of atmospheric pressure.





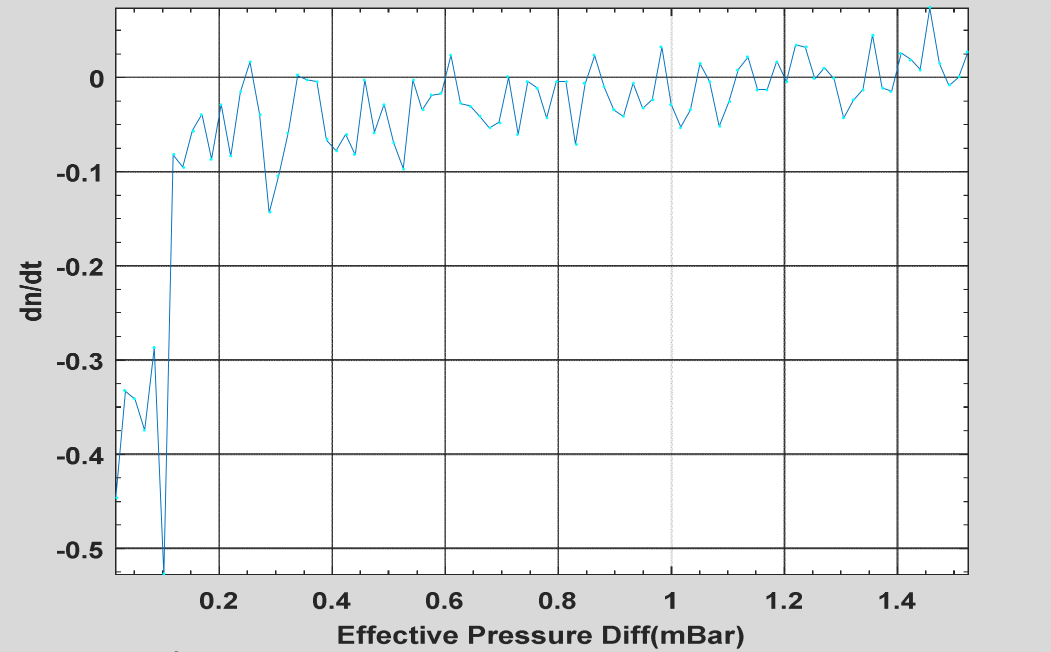


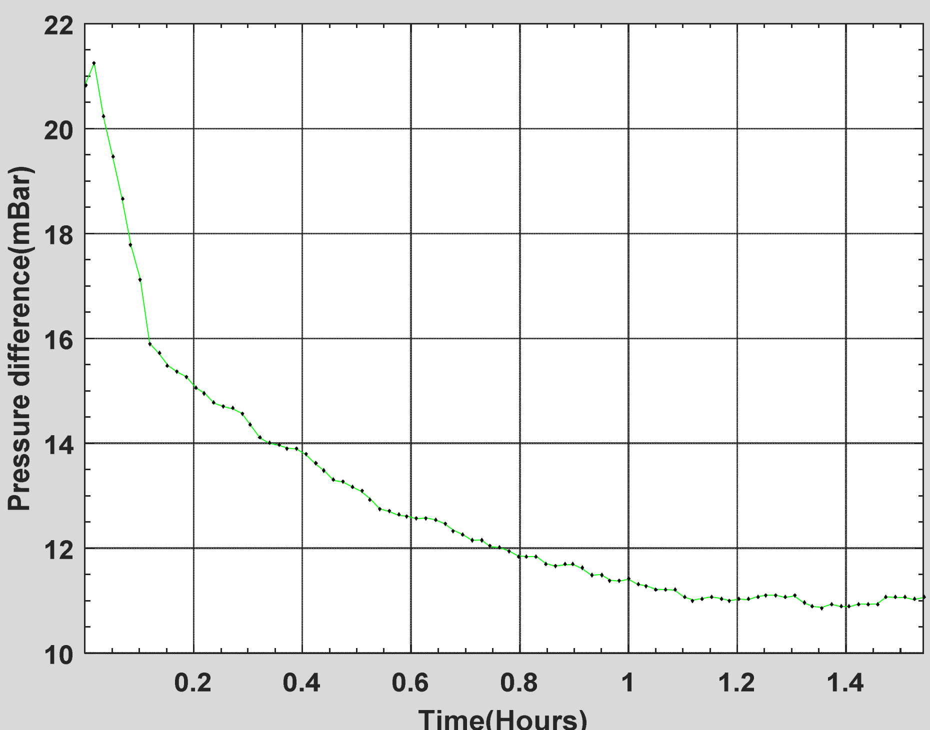
Absolute Leak Rate

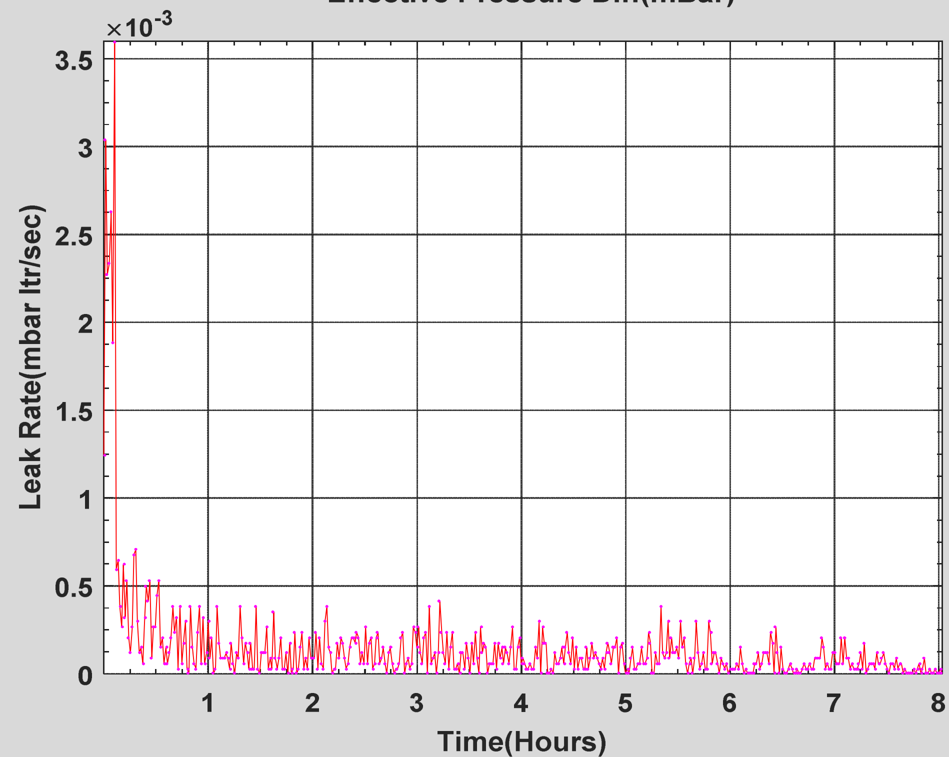
- The leak rate is defined as the pV throughput of a gas through a leak.
- In a system of volume V the leak rate $Q = V.\Delta P/\Delta t$
- Leak Units mbar.l/sec

1 litre will change in pressure by 1 mbar in 1 second

- 1 mbar.l/s = 0.987 Atm. cm^3/s
  = 0.75 Torr.l/s= 0.1 Pa. m^3/s







Aging of gaseous detectors

- **Classical Aging**
  - Study of plasma physics: chemical reactions during avalanche formation leading to conducting/insulating deposits on electrode surfaces.
  - In the Gas Detector community: Radiation Damage is referred to as Aging. Aging of gaseous detectors means degradation of their performance under the exposure to ionizing radiation.
  - DC plasma: Plasma is a mixture of positive ions, neutral species, and chemically active free radicals
  - Polymerization: process by which some radicals associated with a reaction chain form a very large molecule, of high molecular weight.
  - Created polymer has high adhesion probability to surfaces.

Aging (a complex phenomenon)

- **The origin of impurities is diverse and depends on many parameters**
  - Aging depends on the gas quality and may be enhanced by the presence of impurities in the gas itself, which are transported by the gas flow and deposited due to electrostatic forces.
  - Outgassing from the materials used in the construction of the detector itself (e.g. glues, spacers)
  - Contamination of the detector during the assembly process (possible sources are clothes; hair, make-up, fingernail and fingerprints are a source of oil and particular, many creams and cosmetics contain silicones; soldering equipment that requires heating of volatile fluxes.)
  - Pollutants released by materials used to build gas system and tubing (potential polluting sources : plastic piping; valves and cylinder pressure regulators, etc.).

- Nonmetallic materials outgas vapours which, transported by the gas flow and may be deposited directly on the surface of the electrodes, and favour the polymerization of radicals in the avalanche plasma.

Potential aging effects :

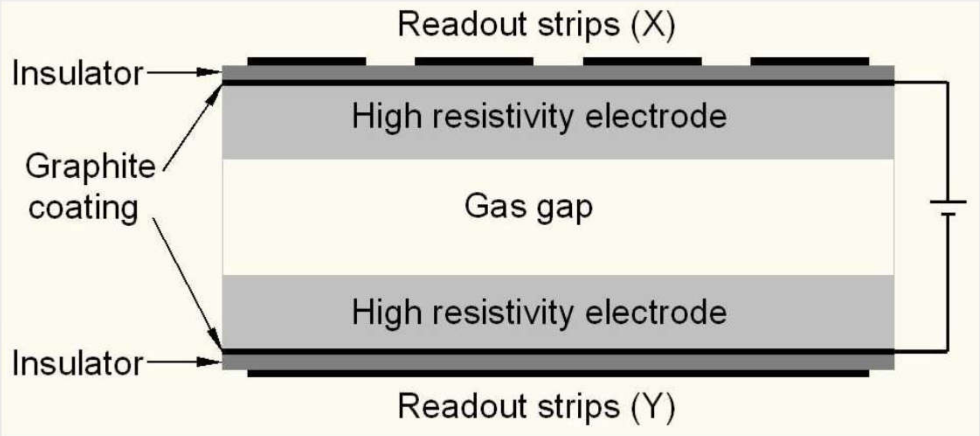
-
- **Deterioration in Performance**:
  - Decreases and non-uniformity of the gas gain
  - Loss of efficiency
  - Worsening of energy resolution
  - Excessive currents
  - Distortion of the pulse height distribution
  - Electron Emission (Malter Effect)
  - Self-sustained discharges
  - Sparking
  - The etching of the surfaces/ Change of surface quality

- Shortened the lifetime of the detector

RPC detectors at DU

- **Resistive Plate Chambers (RPCs): gaseous parallel plate detectors**
  - Exp: INO-ICAL
  - Detector: RPC (Large areas at low production cost, high time resolution, good spatial resolution)
  - Material: Glass
  - Layout: Single-gap
  - Gas system operation: Open-loop mode (Gas mixture exiting the detectors is sent into the atmosphere, few parameters to monitor)
  - Gas Mixture: R134a ($C_2H_2F_4$) /iC_4H_{10}/SF_6 (non-flammable mixture)
  - Gas gap: 2mm
  - Particle: muons
  - Use of Fluorine in the mixture: Yes

Resistive Plate Chamber (RPC) Detector

➢ 

➢ The Resistive Plate Chamber (RPC) was developed in 1981 by R. Santonico and R. Cardarelli.

➢ RPC detector consists of two parallel plate electrodes of high resistivity, such as glass or bakelite (phenolic resin).

➢ On top of the plates a graphite coating is applied in order to distribute the high voltage on the electrodes which leads to a uniform electric field in the gas gap.

➢ The readout is performed by means of copper strips running along the whole length of the chamber on both sides of the gap, but perpendicular, allowing read out of the x- and y- coordinate position of a traversing particle.

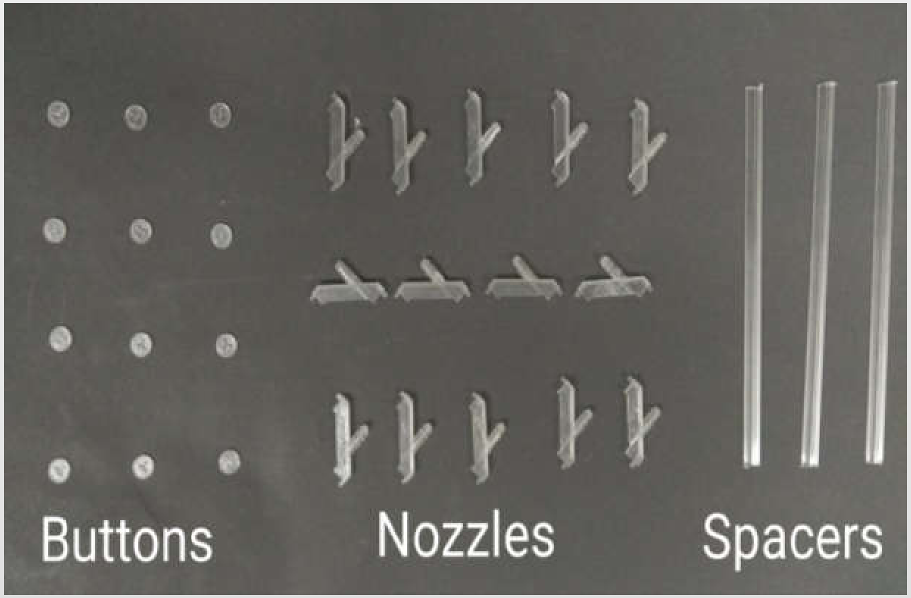
➢ Working Principle: the passage of a charged particle through the gas gap produce primary ionization cluster, which under the effect of electric field develop avalanches in the gas. This exponential avalanche growth induces signal on the external readout electrodes.

Aim of the present study

- To identify all the impurities and dangerous radicals produced due to outgassing when RPC was operated in the cosmic stand .
- To estimate the outgassing due to the various materials used in the construction of INO RPCs like glues, buttons spacers, frames, etc.
- Selection of materials and gases usable for chamber construction and operation with acceptable outgassing properties.
- To check the gas mixture quality and purity.
- To check the fluoride ion production due to $C_2H_2F_4$ molecule breaks under the effects of cosmic muons and electric field.
- The identification of all the extra-components produced in the mixture during the RPC operation.

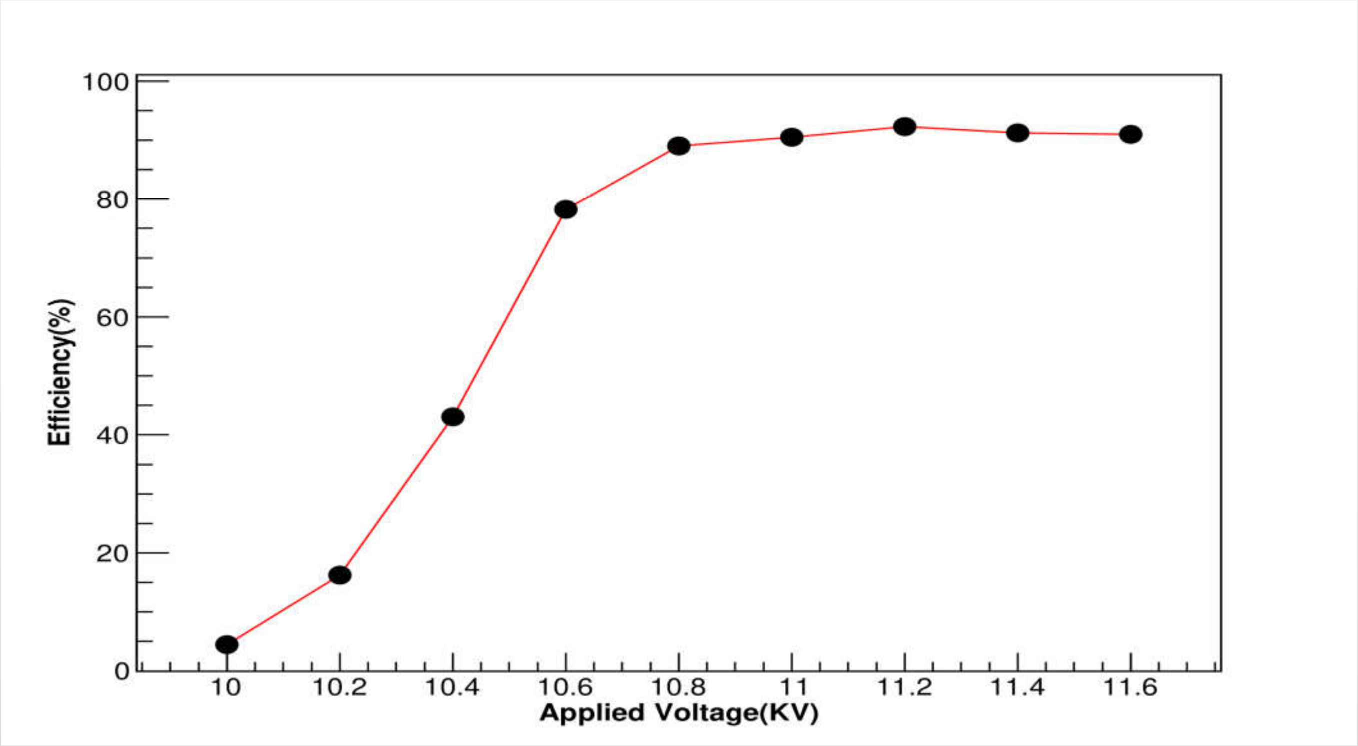
Materials used in the fabrication of RPC

➢



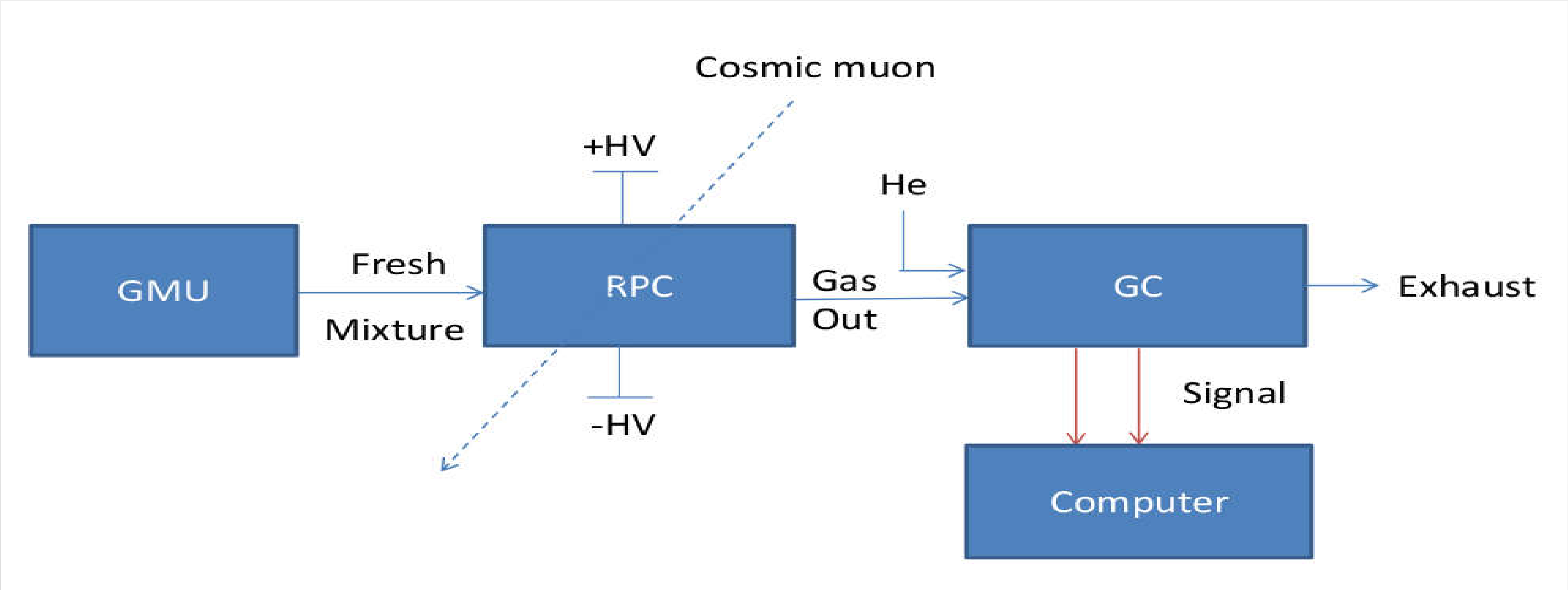
- ➢ The buttons, spacers and nozzles are made of polycarbonate materials of high resistivity (non- reactive nature?)
- ➢Buttons : To ensure the uniformity of the gap
- ➢Spacers : To seal the sides and form an airtight gas volume
- ➢Nozzles : In order to make gas flow inside the chamber
- ➢ Glue used : 3M scotch-weld epoxy adhesive DP-125

Efficiency



- ➢ Working Point: the lower the better (Electric field play a key role: The impurities in the gas, which are transported with the gas flow may directly deposit on the surface of electrodes due to electrostatic forces)
- ➢ Temperature: the lower the better 20°C to 22°C (At high operating temperature, the chamber draw more current)
- ➢ Relative humidity: best value 35-40% for stable resistivity and low leakage current.

Experimental Test Facilities

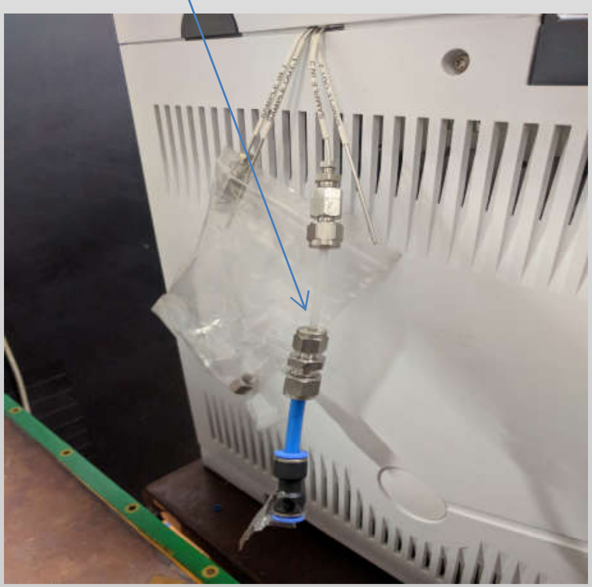


- Typical test stand for detector aging studies
- Used Technologies to study gas aging : GC (Gas Chromatography)
- The gas mixture flows into the RPC detector, and then enters GC chamber.
- A gas chromatograph (GC) is used for analysing the gas as it flows out of the test chamber.

Gas Analysis

- The study was performed using a Gas Chormatograph (Agilent 7890B GC System)
- Gas Chromatograph: it separates and quantifies the gas mixture components
- Sensitivity ~ ppm
- Oven with a capillary column capable of separating gas substances depending on their interaction properties with the column.

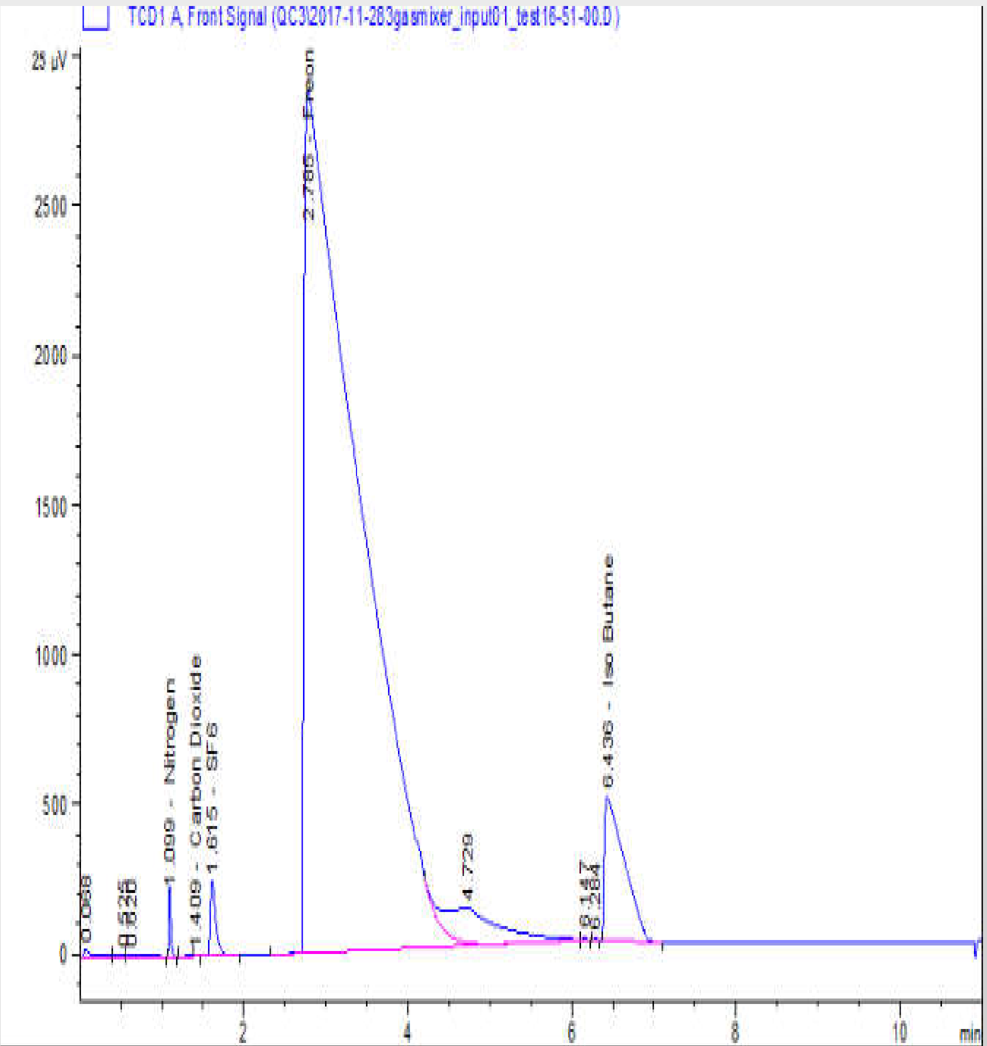
Injector



Column

Input Reference Gas

Gas chromatogram of the fresh gas mixture before it enters the RPCs



Signal 1: TCD1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [25 µV*s]	Area %	Name
1	0.068	BV	0.1022	202.78888	0.13610	?
2	0.525	VV	0.1188	49.39601	0.03315	?
3	0.620	VB	0.2691	112.08032	0.07522	?
4	1.099	BB	0.0302	402.04178	0.26982	Nitrogen
5	1.409	BV E	0.0297	2.25179	0.00151	Carbon Dioxide
6	1.615	VB R	0.0730	1177.42212	0.79019	SF6
7	2.785	BV R	0.5708	1.33301e5	89.46082	Freon
8	4.729	VV E	0.5897	5515.22266	3.70137	?
9	6.147	VB E	0.0567	42.86560	0.02877	?
10	6.284	BB	0.0480	28.82445	0.01934	?
11	6.436	BB	0.2358	8171.00293	5.48372	Iso Butane

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Signal 1: TCD1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [25 µV*s]	Area %	Name
1	0.070	BV	0.1053	187.14032	0.12347	?
2	0.519	VV	0.1734	69.49187	0.04585	?
3	0.608	VB	0.2629	113.18419	0.07467	?
4	1.098	BB	0.0276	164.10640	0.10827	Nitrogen
5	1.399		0.0000	0.00000	0.00000	Carbon Dioxide
6	1.611	BB	0.0715	1208.90442	0.79758	SF6
7	2.769	BV R	0.5739	1.36843e5	90.28315	Freon
8	4.733	VV E	0.5790	4684.69629	3.09075	?
9	6.145	VB E	0.0533	41.42694	0.02733	?
10	6.281	BB	0.0483	28.81214	0.01901	?
11	6.434	BB	0.2355	8230.20215	5.42992	Iso Butane

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Day 1

After assembling a new RPC detector, the input gas flow through the detector in order to check if some contamination occurs during the assembly process.

Without applying the HV

Peak #	RetTime [min]	Type	Width [min]	Area [25 µV*s]	Area %	Name
1	0.073	BV	0.0952	122.57359	0.07966	?
2	0.617	VB	0.4663	243.13731	0.15801	?
3	1.105	BB	0.0278	199.66101	0.12975	Nitrogen
4	1.415	BB	0.0481	4.54616	0.00295	Carbon Dioxide
5	1.619	BB	0.0706	1174.70251	0.76340	SF6
6	2.780	BV R	0.5722	1.38312e5	89.88469	Freon
7	4.739	VV E	0.5921	5503.11279	3.57630	?
8	6.156	VB E	0.0572	43.93182	0.02855	?
9	6.293	BB	0.0487	29.26241	0.01902	?
10	6.447	BB	0.2209	8244.23730	5.35767	Iso Butane
Totals :				1.53877e5		

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No extra-component observed

After applying the HV = 11 KV

Peak #	RetTime [min]	Type	Width [min]	Area [25 µV*s]	Area %	Name
1	0.069	BV	0.0903	142.30231	0.09322	?
2	0.288	VV	0.1255	63.61525	0.04167	?
3	0.466	VV	0.1527	58.85052	0.03855	?
4	0.619	VB	0.2708	112.52542	0.07371	?
5	1.109	BB	0.0304	202.80676	0.13285	Nitrogen
6	1.421	BV E	0.0310	2.99421	0.00196	Carbon Dioxide
7	1.629	VV R	0.0745	1232.73682	0.80750	SF6
8	2.803	BV R	0.5672	1.36250e5	89.25066	Freon
9	4.738	VV E	0.5869	6210.97705	4.06850	?
10	6.162	VB E	0.0577	46.13367	0.03022	?
11	6.300	BB	0.0486	29.31635	0.01920	?
12	6.452	BB	0.2237	8307.69922	5.44196	Iso Butane

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An extra-component at RetTime 0.288 appear

Day 2-3

Day 2

Peak #	RetTime [min]	Type	Width [min]	Area [25 µV*s]	Area %	Name
1	0.070	BV	0.0883	136.41861	0.08786	?
2	0.290	VV	0.1175	55.55739	0.03578	?
3	0.522	VV	0.1371	72.12230	0.04645	?
4	0.618	VB	0.2670	124.20673	0.07999	?
5	1.106	BB	0.0324	579.51422	0.37322	Nitrogen
6	1.417	BV E	0.0334	3.24196	0.00209	Carbon Dioxide
7	1.624	VV R	0.0719	1235.16785	0.79547	SF6
8	2.021	VB E	0.0879	2.28505	0.00147	?
9	2.790	BV R	0.5805	1.38660e5	89.29958	Freon
10	4.734	VV E	0.5939	5977.15479	3.84938	?
11	6.158	VB E	0.0552	45.12321	0.02906	?
12	6.295	BB	0.0482	28.93868	0.01864	?

A new peak at RetTime 2.02 min was observed with 0.001 area % in mixture.

Day 3

Peak #	RetTime [min]	Type	Width [min]	Area [25 µV*s]	Area %	Name
1	0.072	BV	0.0937	124.56626	0.07967	?
2	0.622	VB	0.4743	274.31912	0.17544	?
3	1.107	BB	0.0313	392.30258	0.25090	Nitrogen
4	1.399		0.0000	0.00000	0.00000	Carbon Dioxide
5	1.623	BB	0.0716	1165.35645	0.74532	SF6
6	2.784	BV R	0.5830	1.40848e5	90.08046	Freon
7	4.739	VV E	0.5667	5132.21191	3.28236	?
8	6.160	VB E	0.0570	43.87880	0.02806	?
9	6.297	BB	0.0481	29.34905	0.01877	?
10	6.450	BB	0.2254	8347.95215	5.33902	Iso Butane
Totals :				1.56357e5		

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Disappearance of the peaks. No new peak was observed.

Day 7

Day 7

Peak #	RetTime [min]	Type	Width [min]	Area [25 µV*s]	Area %	Name
1	0.071	BV	0.0847	119.48984	0.07637	?
2	0.614	VV	0.3392	218.58800	0.13971	?
3	0.724	VV	0.0972	42.39020	0.02709	?
4	0.879	VB	0.1741	47.22784	0.03019	?
5	1.093	BB	0.0317	515.37524	0.32941	Nitrogen
6	1.399		0.0000	0.00000	0.00000	Carbon Dioxide
7	1.602	BV E	0.0802	1402.33276	0.89632	SF6
8	2.755	VV R	0.5851	1.40214e5	89.61916	Freon
9	4.715	VV E	0.5676	5479.14355	3.50205	?
10	6.137	VB E	0.0536	42.41015	0.02711	?
11	6.273	BB	0.0486	28.99764	0.01853	?
12	6.427	BB	0.2317	8345.41895	5.33406	Iso Butane

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Extra peaks at RetTime 0.72 min & 0.88 min

Day 8

Peak #	RetTime [min]	Type	Width [min]	Area [25 µV*s]	Area %	Name
1	0.063	BV	0.0902	569.48962	0.36843	?
2	0.508	VV	0.2716	540.23114	0.34950	?
3	0.599	VB	0.2061	356.45599	0.23061	?
4	1.096	BB	0.0302	469.37225	0.30366	Nitrogen
5	1.401	BV E	0.1058	17.77075	0.01150	Carbon Dioxide
6	1.602	VB R	0.0734	1225.60181	0.79291	SF6
7	2.748	BV R	0.5700	1.37156e5	88.73347	Freon
8	4.719	VV E	0.5897	5808.86084	3.75806	?
9	6.135	VB E	0.0556	41.57221	0.02690	?
10	6.272	BB	0.0483	29.34931	0.01899	?
11	6.425	BB	0.2412	8356.04688	5.40597	Iso Butane

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Again Disappearance of the extra peaks

Some Extra-components

Day 65

1	0.055	BV R	0.0909	1853.07947	1.20085	?
2	0.271	VV E	0.1257	5.98834	0.00388	?
3	0.511	VV X	0.0489	1.90352	0.00123	?
4	0.631	VV X	0.0480	1.36829	0.00089	?
5	0.772	VB X	0.1839	84.31316	0.05464	?
6	1.095	BB	0.0302	153.45956	0.09945	Nitrogen
7	1.400	BV E	0.0706	22.67855	0.01470	Carbon Dioxide
8	1.605	VB R	0.0718	1237.51855	0.80195	SF6
9	2.768	BV R	0.5638	1.37258e5	88.94724	Freon
10	4.713	VV E	0.5517	5302.96777	3.43648	?
11	6.146	VB E	0.0527	48.43290	0.03139	?
12	6.284	BB	0.0481	27.90959	0.01809	?

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Day 75

Peak #	RetTime [min]	Type	Width [min]	Area [25 µV*s]	Area %	Name
1	0.067	BV	0.0830	148.80171	0.09529	?
2	0.520	VV	0.2619	139.20985	0.08914	?
3	0.623	VV	0.1357	65.94357	0.04223	?
4	0.774	VB	0.1599	65.42300	0.04189	?
5	1.097	BB	0.0297	165.97977	0.10629	Nitrogen
6	1.399		0.0000	0.00000	0.00000	Carbon Dioxide
7	1.610	BB	0.0720	1208.37988	0.77379	SF6
8	2.769	BV R	0.5865	1.40346e5	89.87155	Freon
9	4.718	VV E	0.5601	5571.80322	3.56794	?
10	6.150	VB E	0.0533	55.00032	0.03522	?
11	6.289	BB	0.0479	26.96803	0.01727	?
12	6.442	BB	0.2237	8369.38574	5.35939	Iso Butane

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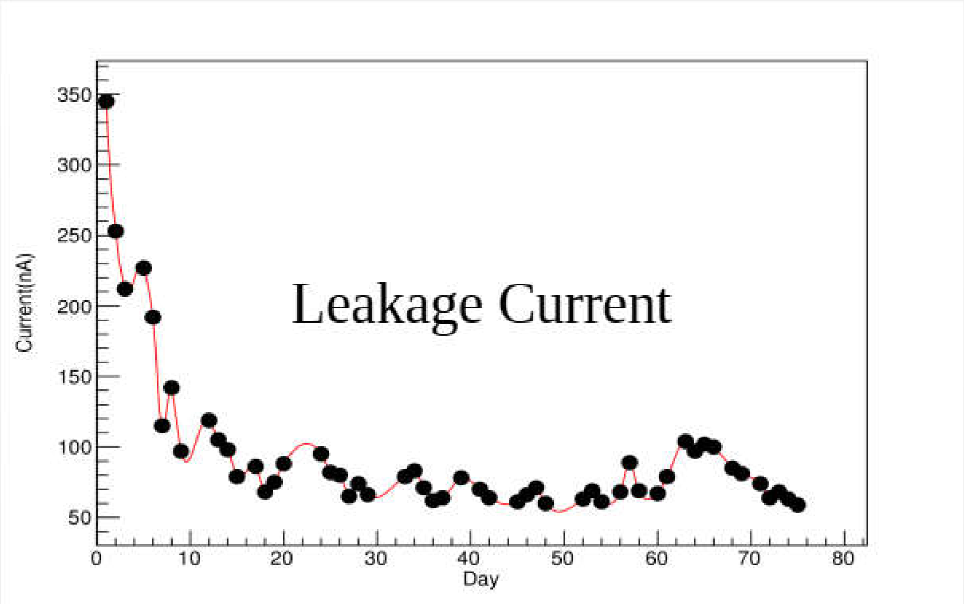
Extra peaks at RetTime 0.77 min with 0.055 area % in mixture

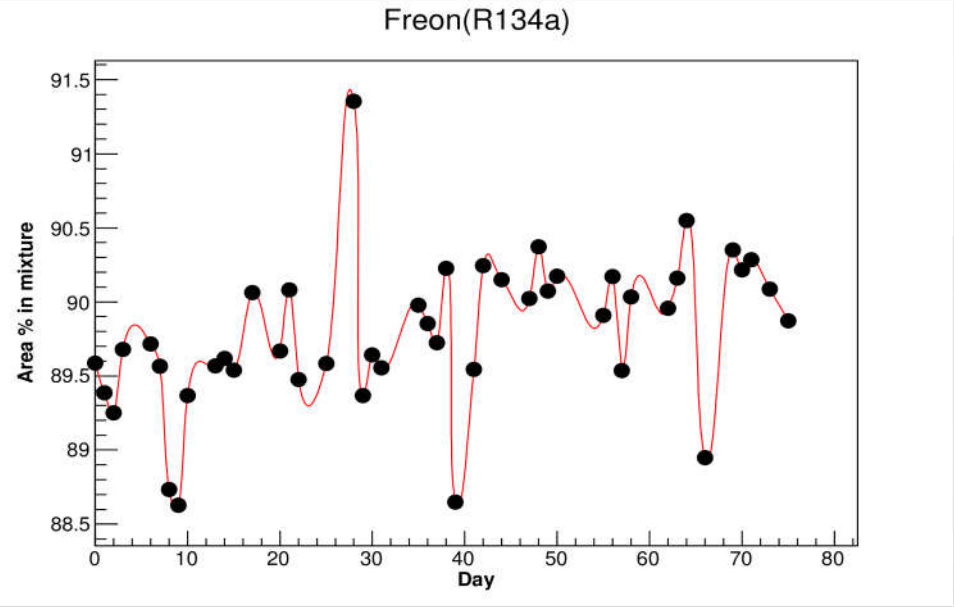
Extra peaks at RetTime 0.77 min with 0.042 area % in mixture

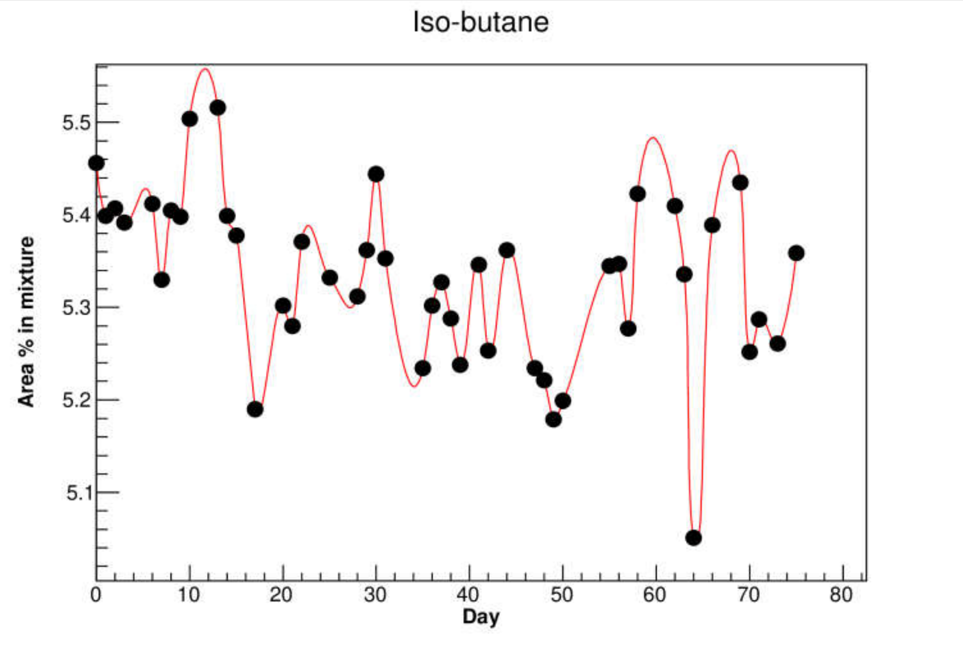
Extra Peaks:

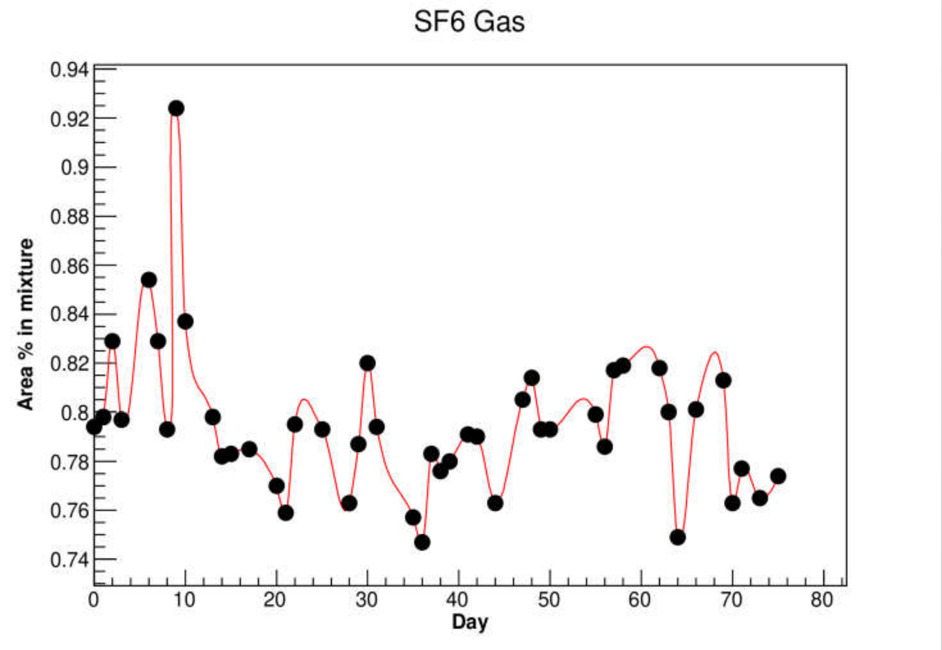
- Day 13 : Peak at RetTime = 0.69 min with 0.198 area percentage in gas mixture.
- Day 27 : Peak at RetTime = 0.77 min with 0.121 area percentage in gas mixture.
- Day 41 : Peak at RetTime = 0.75 min with 0.042 area percentage in gas mixture.
- Day 46 : Peak at RetTime = 0.66 min with 0.05 area percentage in gas mixture
- Day 47 : Peak at RetTime = 0.71 min with 0.028 area percentage in gas mixture.
- Day 65 : Peak at RetTime = 0.77min with 0.055 area percentage in gas mixture.
- Day 75 : Peak at RetTime = 0.77 min with 0.042 area percentage in gas mixture.

Some general parameters

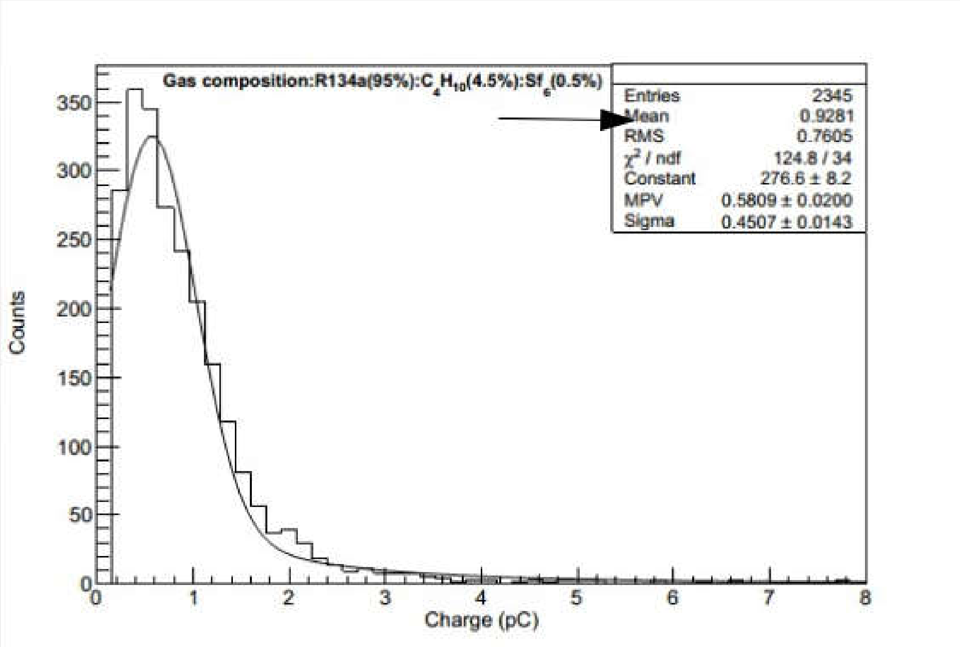


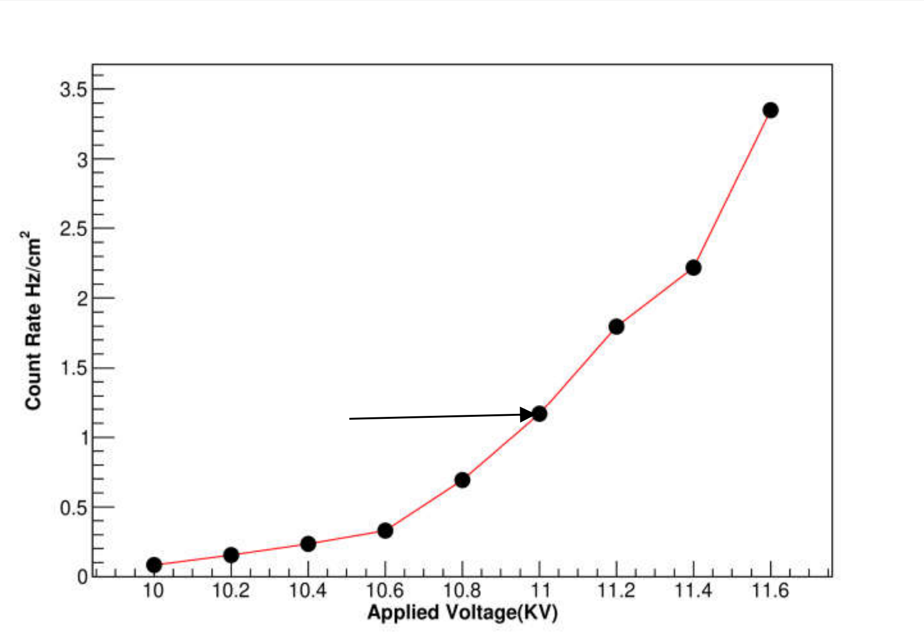






Total accumulated charge





- Count rate at 11.0 KV = 1.2 Hz/cm²
- Average charge per event delivered in the gas at the working point = 0.92 pC (~ 1 pC safety factor)
- Total charge in one day = 1 X 1.2 X 3600 X 24 pC/cm²
- Total charge in 75 days = 1 X 1.2 X 3600 X 24 X 75 pC/cm²
  = ~ 8 μC/cm²

Conclusions

- **A quantitative estimation of the leak test of an RPC is obtained without using conventional manometer.**

- **We demonstrated that materials such as polycarbonate buttons, spacers and the 3M scotch-epoxy adhesive DP-125 produces no significant pollutant outgassing at room temperatures, and thus do not affect the lifetime of gas detectors.**

- **These materials are suitable for the assembly of RPC detectors in the cosmic experiments.**

- **The result of last 75 days shows no permanent extra component in the return mixture from the RPC detector in the open loop gas system and comic background.**

- **But some extra component has been observed, but their occurrence is random in time and also they vary in area percentage of the mixture (Need more study).**

References:

- [1] M.Capeans et al., About aging of gas detectors: a compilation of some validation studies carried out for LHC, RD51 Note-2009-002.
- [2] M.Titov, Radiation damage and long-term aging in gas detectors, arXiv:physics/0403055v2, 2004.
- [3] M.Capeans et al., Recent aging studies for the ATLAS transition radiation tracker, IEEE Trans. Nucl. Sci. Vol.51, 3, 2004.
- [4] M.Capeans et al., RPC performances and gas quality in a closed
- loop gas system for the new purifiers configuration at LHC experiments, JINST 8 T08003, 2013.
- [5] J. Va'vra, Summary of session 6: aging effects in RPC detector,
- Nucl. Instr. and Meth A 515(2003)354.
- [6] Carsten Niebuhr, Aging Effects in Gas Detectors, Nucl. Instr. and Meth A 566(2006)118.
- [7] R. Bouclier et al., Effects of outgassing from some materials on gas chamber ageing, Nucl. Instr. and Meth A 350(1994)464.

- Back up

Aging in RPC

- GC : Polar polyethylene glycol deactivation layer
- It provide optimum wettability for polar compounds
- Ultra Inert gold seals
- Inside GC column: Fused silica surface (stationary phase)
- Outer coated by Polymide

- Very clean gas mixtures, no hydrocarbons in gas mixtures, no use of silicone
- Under the effects of background radiation and electric field, $C_2H_2F_4$ molecule breaks into fluorine radicals
- Creation of F - radical free

Working Gas

- Most RPCs operate in avalanche mode due to the much higher rate capability compared to streamer mode. In order to stay in the region of avalanche mode, streamers must be suppressed.
- The working gas composition used in RPCs has a large impact on the detector performance . Noise rates, sensitivity, spatial resolution and power consumption can all be influenced by the choice of gas mixture.

- 1.) Tetrafluoro ethane C2H2F4 (R134a) : ionization
- fairly dense gas resulting in high primary ionization which allow good detectable signal and to achieve high efficiency, high drift elecron velocity in C2H2F4 allowsgood time characteristics.
- Other advantages of tetrafluorethane are that it is non-flammable and environmental safe.
- 2.) C4H10 isobutane: γ absorption
- Butane supress creation of after pulse by absorping photons which come from gas ion and electron reconbination. Thus ensures the locality of the discharge by limiting the formation of secondary avalanches far from the primary ones.
- 3.) SF6 : quench
- Highly electronegative gas, which prevents avalanche size from spreading transversely by absorping free electrons in the gas during avalanche development.