Fluorine impurities in Triple-GEM detectors with CF_4-based gas mixtures

M. Corbetta, R. Guida, B. Mandelli

RD51 Mini Week, 17/02/2021

EP-DT
Detector Technologies

Outline

- ➔ Impurities and gas recirculation
  - ◆ Fluorine-based impurities
- ➔ Experimental setup
  - ◆ Gamma Irradiation Facility (GIF++)
  - ◆ Fluoride ion measurement
- ➔ Results of Process Characterization
  - ◆ Effect of CF4 concentration variations
  - ◆ Effects of charge density
  - ◆ Effects of input gas flow rate
- ➔ Results of Closed Loop operation
- ➔ Run 2 measurements on LHCb GEM
- ➔ Conclusions

Gas mixture impurities

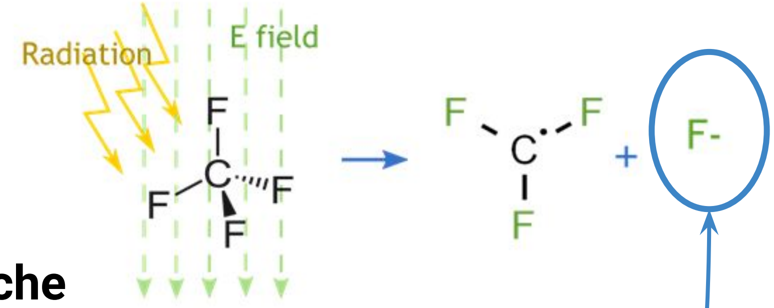
Gas mixture : primary element influencing gas detectors performance

- Impurities are a key factor compromising gas mixture quality
- Common gas system impurities: N_2, O_2, H_2O
  from air-intake of gas system elements or detectors
- *Freon-component can create fluorine-based impurities*
  > phenomenon well known and studied for RPC detectors
- Risk: compromise detector performance, accelerate aging phenomena

Gas recirculation

- Reduction of gas consumption and emission
- Impurities accumulation in the gas system

Fluorine-based Impurities in Triple-GEMs



- **CF_4 can be present in Triple-GEMs mixture**
  to enhance time resolution ($Ar/CO_2/CF_4$ 45/15/40)
- Radiation + E field = high charge density
- **CF_4 easily breaks up in the electron avalanche**
- Highly reactive products: F-, CF_3+, CF_2+, ...
  - Polymerized deposits (HF acid, if H_2O present)
  - Material etching (GEM foils, readout electrode)

- Factors affecting impurities production + entity of aging effects
  - Charge density (incoming radiation, electric field)
  - Gas mixture composition (CF_4 concentration)
  - Gas flow rate in chamber volume
  - Progressive accumulation in gas recirculating systems

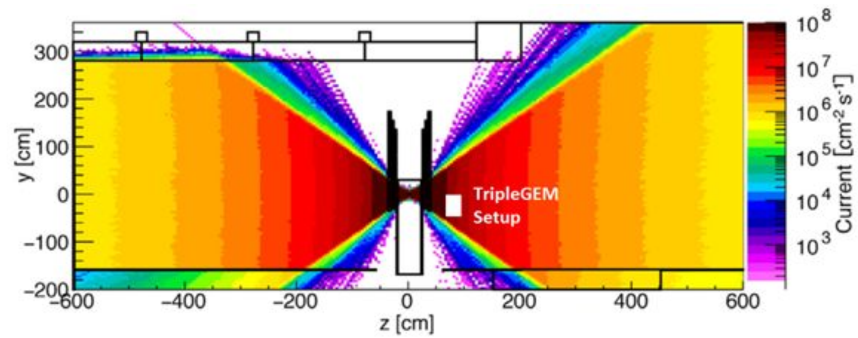
- *Fluoride ions = quantitative indication on created impurities*

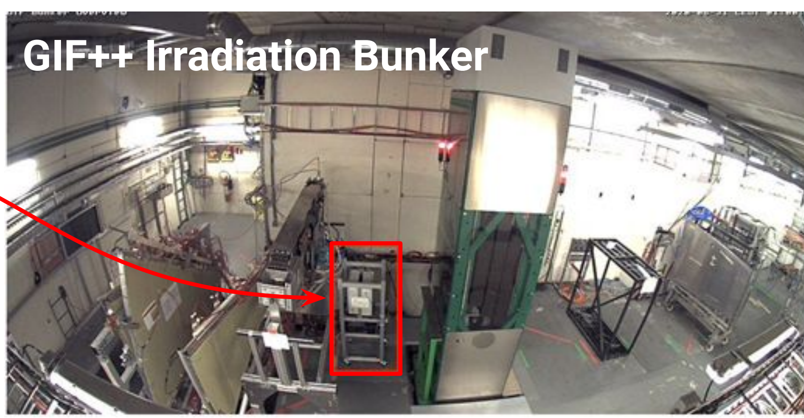
Experimental Setup: Gamma Irradiation Facility (GIF++)

Dedicated test zone for large-area muon chambers,
for performance characterization and aging tests

- **Irradiation provided by ^{137}Cs source**,
  662 keV photons, activity 14 TBq
- Variable source intensity thanks to integrated absorption filter system
- *Dose rate suitable to mimic high-rate radiation of HL-LHC Phase (1G/h at 1m)*

- Triple-GEM Gas R&D Setup
  - Two 10x10 cm^2 detectors (gaps 3-1-2-1 mm)
  - Downstream at 1 m from the source
  - Nominal photon current 50 x 10^6 Hz/cm^2 (ABS 1)





Experimental Setup:
Gas system and data acquisition

Small replica of LHC gas system (open mode/closed loop)

- Three-component mixer module
- Ar/CO_2 or $Ar/CO_2/CF_4$
- LHC Purifier module for O_2, H_2O removal

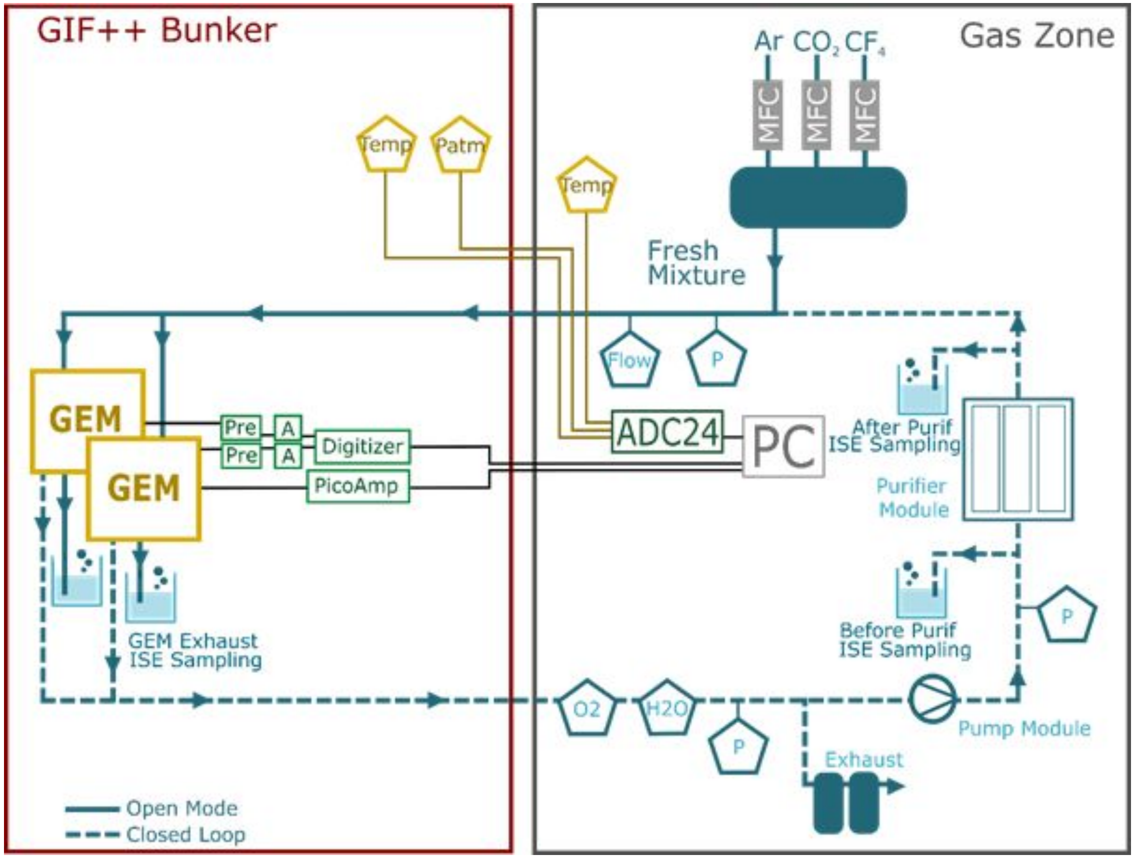
Sensors + ADC data logger

- Env. parameters (Patm, T)
- Gas parameters (flow, P)

Triple-GEMs performance monitored collecting detector current

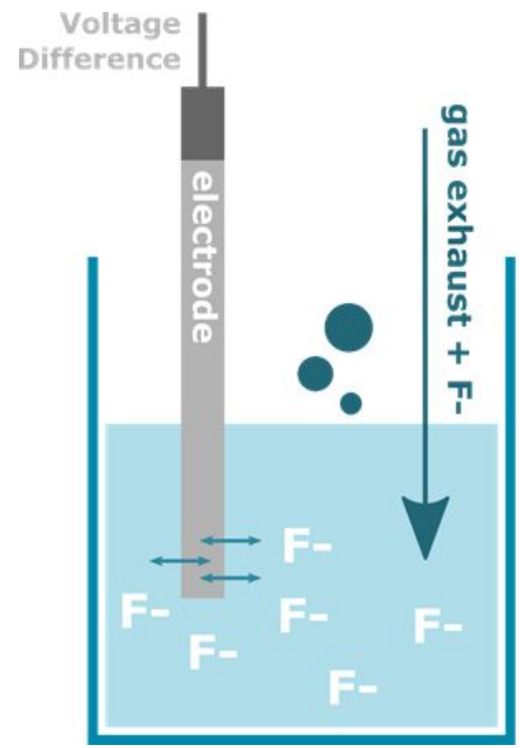
Fluoride impurities measured from gas sampling points along the gas system

- Detector exhaust, before and after purifier



Fluoride Ion measurement

- Ion Selective Electrode station (ISE),
  electrodes specific for Fluoride ion measurement in solution
- Fluoride ions released into sample solution by bubbled gas
  - Solution composition: ½ distilled H_2O, ½ TISABII alkaline
  - TISABII guarantees stable pH and organic strength



- **Exchange of ions** between solution and solid state organic material of electrode **= voltage difference**
- ***Voltage is proportional to F- concentration***
  in the sample solution, not in the chamber volume

- Electrode detection limit 0.02 ppm
- Estimated Fluoride ion concentration:
  1 ppm in solution = 0.04 ppm in chamber volume

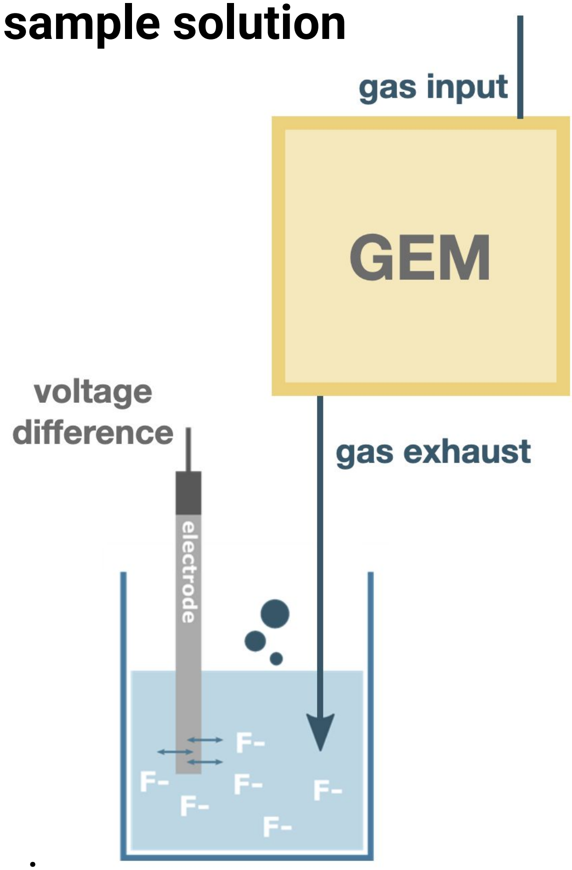
Measurement Method GIF++ Setup

- **Triple-GEMs exhaust gas directly bubbled into ISE sample solution**

- **Fluoride ions** produced in the chamber volume are **deposited into the sampling solution**

- 12-hours long measurement while irradiating
  - *Continuous bubbling for F- accumulation*
  - *Monitoring of Triple-GEM performance with detector current*

- **Variables parameters**
  - CF_4 concentration in standard gas mixture
  - Input gas flow rate
  - High Voltage = Electric field in multiplication region
  - Radiation intensity (change of ABS filter of source)

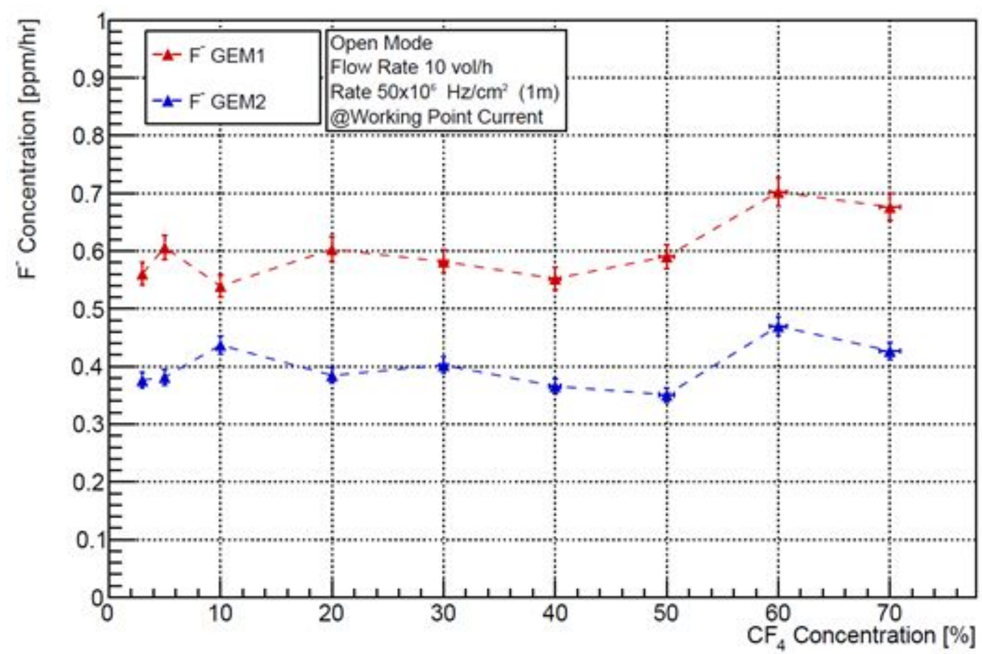


CF_4 concentration GIF++ Setup

- CF_4 concentration in the range 3%-70%, Ar/CO_2 ratio constant (45/15)
- Triple-GEMs irradiated with full ^{137}Cs source (50 x 10^6 Hz/cm^2 γ rate at 1 m)
- HV adjusted to keep detector current constant
  same current >> same charge density

Fluoride ion production rate stable up to 50% CF_4 concentration

- F- production does not strongly depend on the CF_4 content
- Lower CF_4 concentration limit given by Mixer module
  > could be interesting to test the effect of lower fractions

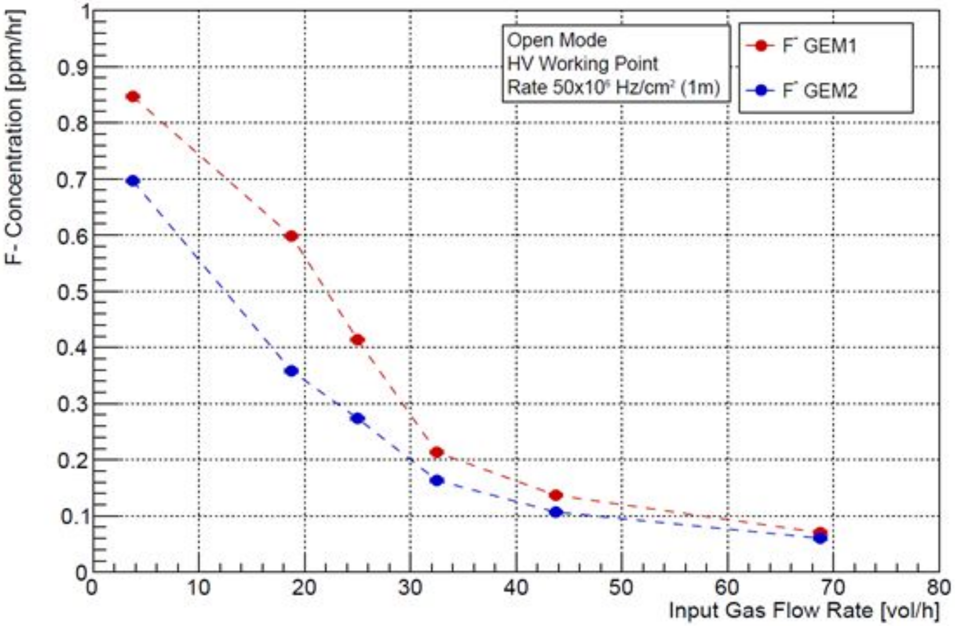


Gas flow rate
GIF++ Setup

- Input gas flow rate varied in the range 5-70 volumes/hour
  > standard Triple-GEM input rate is 10 volumes/hour
- Full exhaust flow bubbled into sampling solution for fixed time
- Fluoride rate normalized by the flow value
  > decouple production from collection

Fluoride accumulation decreases for increasing flow rates

- Higher flows allow faster transit of CF_4 molecules, less time available to break CF_4



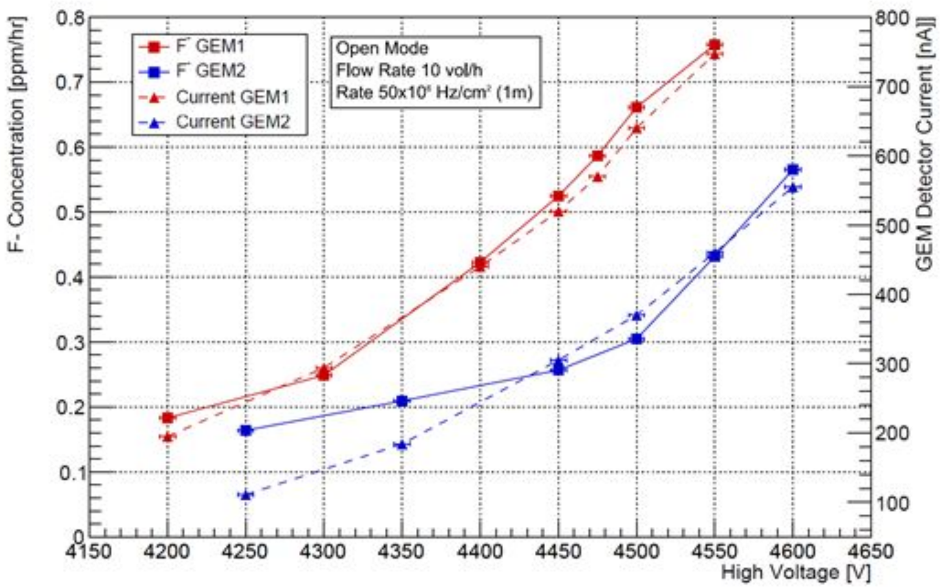
Effect of charge density GIF++ Setup

Charge density in detector volume proportional to detector current
> can vary depending on Electric field or radiation intensity

Variations of Electric field

- Fixed input flow rate (10 vol/h) and radiation rate (50 x 10^6 Hz/cm^2 at 1 m)
- HV increase (within efficiency range, HV work ~4450V)
- Detector current increase
- Fluoride concentration increase

Bigger electron avalanches
due to higher electric field
> more CF_4 molecules are broken



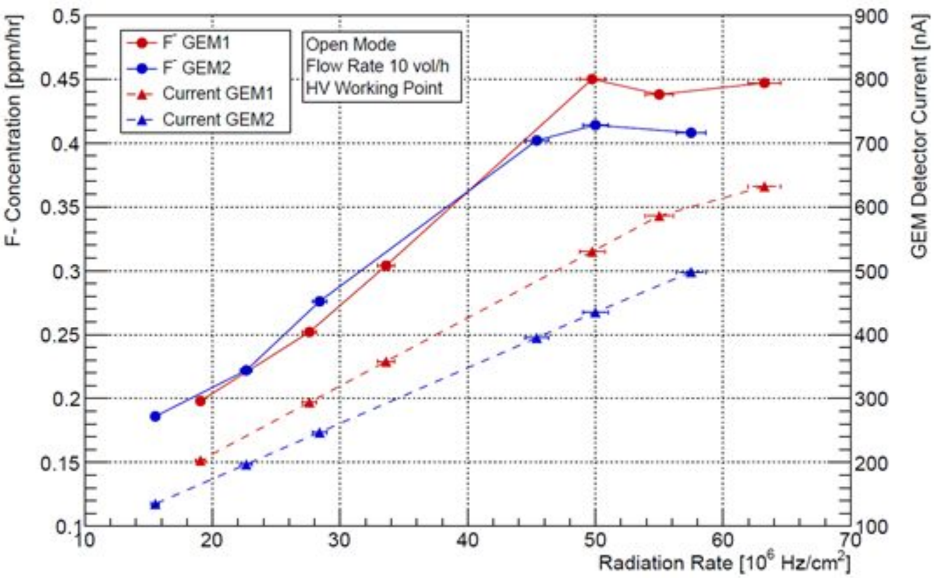
Effect of charge density
GIF++ Setup

Variations of radiation rate

- Fixed input flow rate (10 vol/h) and HV at working point
- Radiation rate increase (15-60 x 10^6 Hz/cm^2) by changing ABS filter
- Detector current increase
- Fluoride concentration **saturation** after ~ 45 x 10^6 Hz/cm^2

Saturation of F- measurement to be further investigated,
could be explained with:

- increase in avalanche size
  VS increase in avalanche number
- Creation of F-, CF_3+
  VS creation of F-, F-, CF_2+
- Production increase but
  F- cannot be extracted
  (saturation in measurement)
- .... any ideas?



Closed Loop operation GIF++ Setup

Triple-GEMs exhaust gas is re-injected into the gas system

- Limit fresh gas consumption
- Accumulation of impurities in the mixture

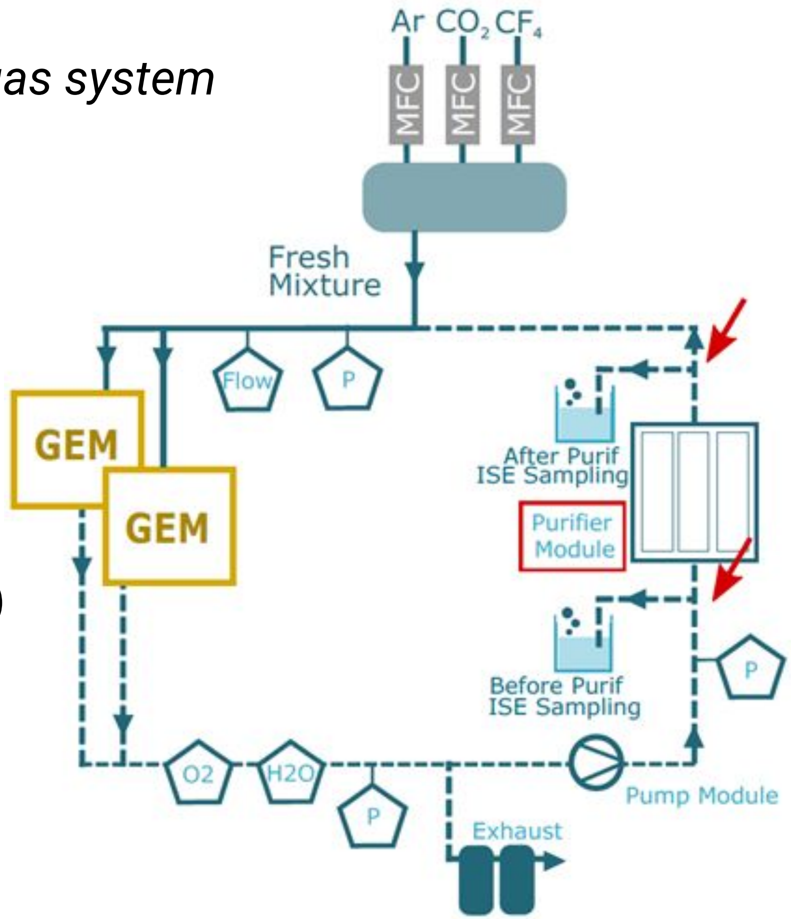
Pump module re-injects the gas, passing through the Purifier Module

- Standard Purifier allows H_2O and O_2 removal

Fluoride accumulation (full irradiation, HV work)

- before purifier: about 1ppm/hour
- **after purifier** module: **0 ppm/hour**

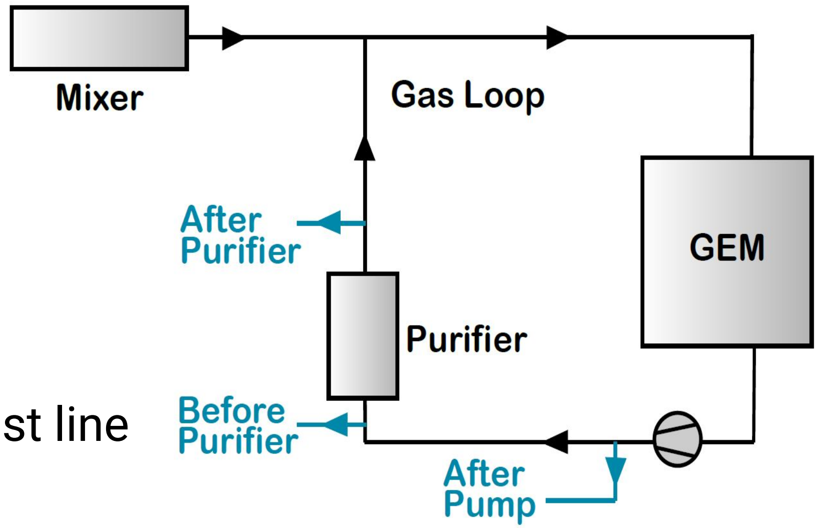
Purifier module traps F- impurities, gas mixture can be safely re-injected



Run 2 LHCb GEM Measurement

Fluoride impurities measured in
LHCb GEM chambers exhaust in Run 2

- Three sampling points,
  after pump (underground),
  before and after the purifier module



Continuous sampling Aug-Dec 2018

- Gas sample 0.5 l/h extracted from exhaust line
- Sampling solution continuously filled
- ISE Station sampling once every 2-3 days

After pump ~ before purifier (separated by 100 mt inox pipe)

After purifier sampling = **0 ppm/hour** along the full period

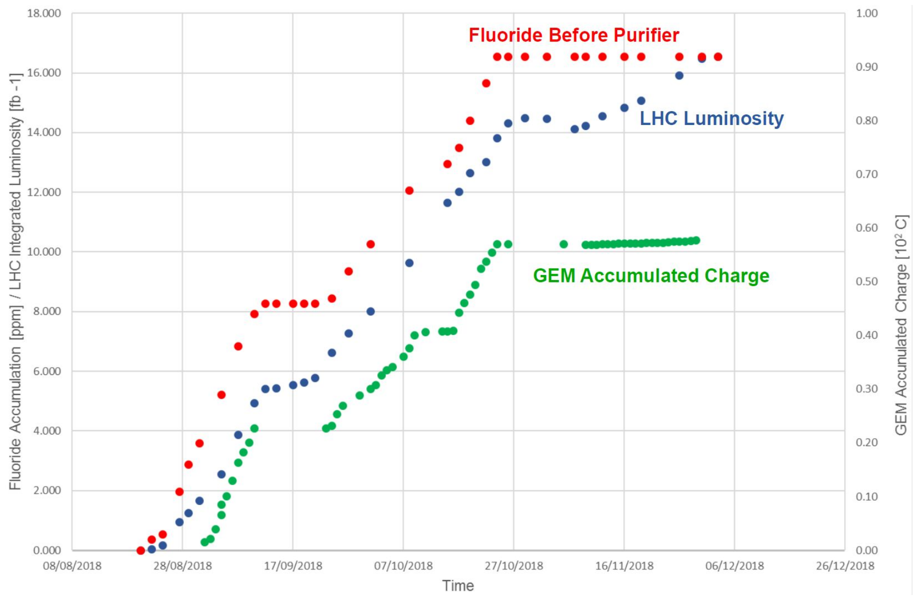
- Confirms the efficiency of purifier module in trapping Fluoride impurities

Run 2 LHCb GEM Measurement

Before purifier sample trend shows **proportionality of Fluoride accumulation with GEM Accumulated charge and LHC Integrated Luminosity**

- GEM accumulated charge ~ integrated detector current increases with the Experiment radiation activity (Luminosity)
- Detector current = charge density in the chamber volume
- Charge density increase yields to fluoride production increase
- Flat periods = tech. stops/ion runs > GEMs off, no accumulation

Coherent results with the ones from GIF++ measurements



Conclusions

Fluoride impurities production process in Triple-GEMs

- CF_4 concentration does not affect the production up to 50%
- High gas flow rates can contribute to decrease impurities concentration

- Electric field and radiation rate are contributing to production increase
  - Irradiation rate saturation effect should be further investigated
  - Proportionality confirmed in LHCb GEM chambers (Run 2)

LHC gas systems with standard purifier module are safe for operation in gas recirculation with CF4-mixture

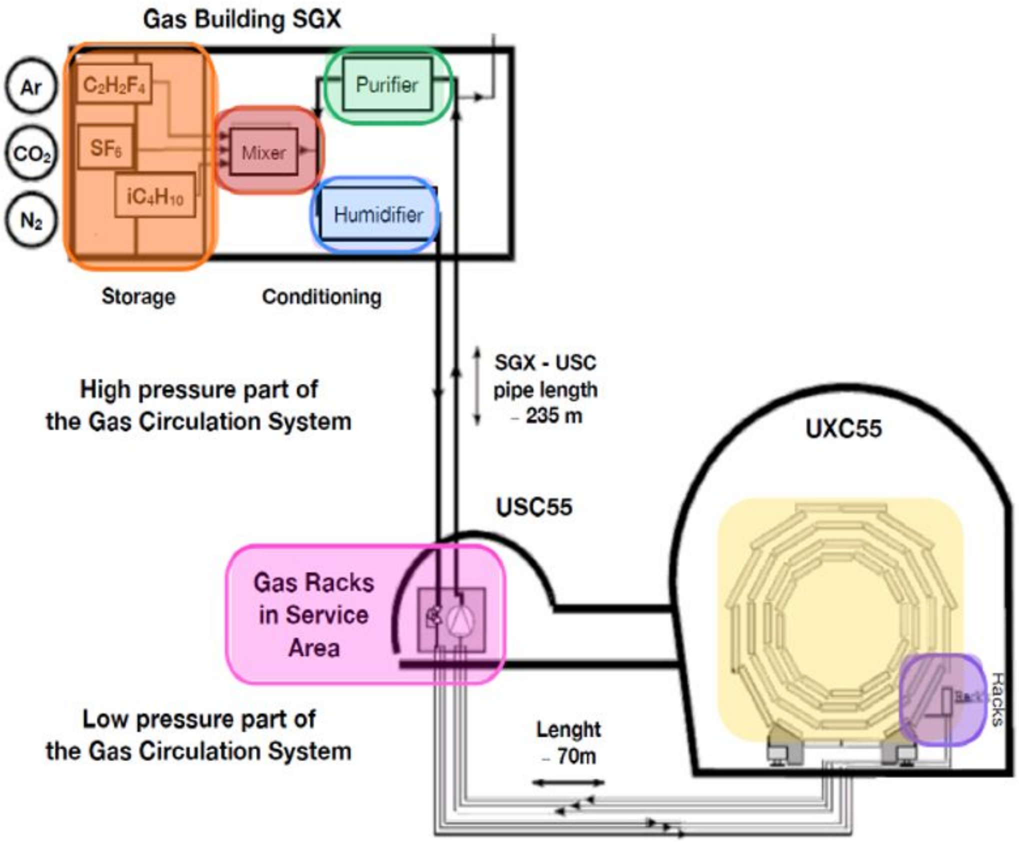
- The O_2/H_2O purifier also traps fluoride impurities
- Results confirmed both in small-replica of gas system at GIF++ and in real-size purifier module of LHCb GEM gas system

Thank you for your attention

Backup

Gas Recirculation in LHC Gas System

- Supply
- Mixer
- Humidifier
- Purifier
- Pump
- Distribution
- Detector



Experimental Setup: Triple-GEM 10x10cm2 prototype

10x10 cm2 Triple-GEMs from CERN PBC Workshop

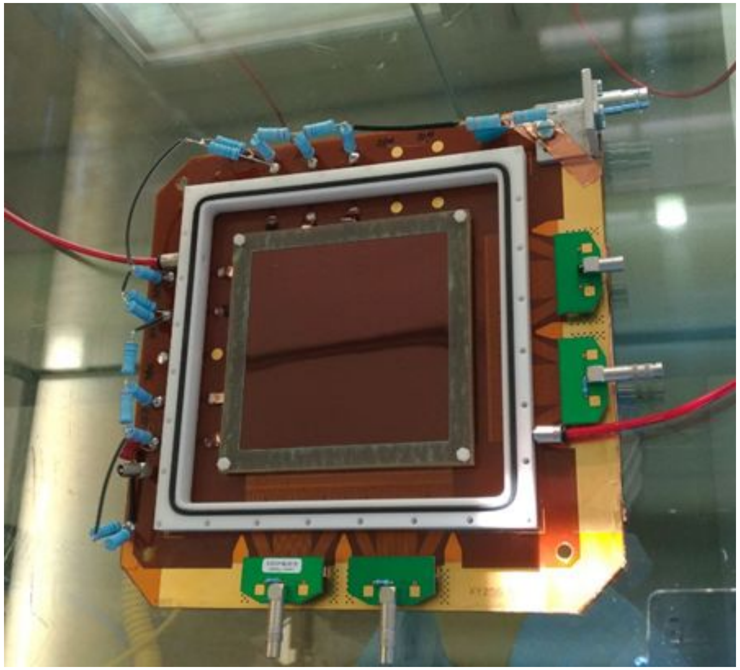
- 3 gem foils, 50µm Kapton (two-side 5µm Copper-clad) + 1 drift foil
- 70µm diameter holes (140µm pitch)
- Gap spacing 3-1-2-1 mm

Cleaning

- Foils and readout board cleaned in several steps
- Baths with KMnO4, H2SO4, Chromic acid and final rinse with deionized water

Assembly procedure

- Foils spaced with fiberglass spacers (EM-470 FR4 base material from EMC)
- Foils stack inside epoxy gas box frame



High Voltage Supply

- single High Voltage line
- custom-made ceramic voltage divider (300 V-400 V per foil)

Experimental Setup: Purifier Module

2 + 1 cartridges (1 liter)

- Molecular Sieve 5Å (MS5A), used for H_2O removal (130 g(H_2O)/kg)
- Catalyst $NiAl_2O_3$, effcient in removing both H_2O and O_2 (15/50 g(H_2O/O_2)/kg)
- Mix of the two materials, allows to operate system when regeneration is needed for the other two

Standard LHC Purifier module, reproduced with smaller columns (LHC column 25 liters)

Fluoride Ion measurement:
Estimation of chamber F- concentration

$$C_{GEM}^{F^-} = \frac{mg_{GEM}^{F^-}}{V_{GEM}} = \frac{mg_{exh}^{F^-}}{V_{exh}} = \frac{mg_{s}^{F^-}}{V_{exh}} = \frac{C_s^{F^-} \times V_s}{V_{exh}}$$

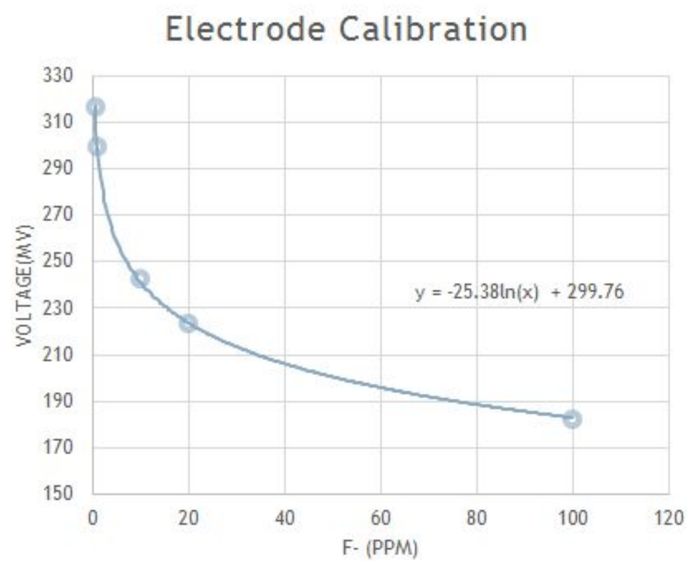
- C $^{F-}$GEM = concentration in ppm in the Triple-GEM chamber
- mg = quantity of Fluoride ion in milligrams
- V = volume in liters
- exh = gas extracted from the sampling point
- s = sample solution
- Gas at chamber exhaust has the same F- concentration as gas in chamber volume
- Equivalence between the F- volume rate in the exhaust flow and F- volume deposited in the sample solution

>> 1 ppm in sample solution = 0.04 ppm in chamber volume

Fluoride Ion measurement: Electrode Calibration

- Electrode calibration realized with Standard solution references: 0.5 ppm, 1 ppm, 10 ppm, 20 ppm, 100 ppm

- Measured voltage difference is linearly proportional to the log of Fluoride concentration
- Calibration is repeated approximately every week to keep stability checked



- Two electrodes were used in parallel
  - Separate (electrode + reference), HANNA Instrument
  - Combined (reference embedded), ThermoScientific
- Equivalent performance in terms of sensitivity Combined electrode reaches equilibrium faster (< 1 minute VS about 5 minutes for 10 ppm)