PROPOSAL FOR A SYSTEMATIC STUDY OF THE CERN CLOSED LOOP GAS SYSTEM USED BY THE RPC MUON DETECTORS IN CMS

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Abstract

We propose to study features, optimization and commissioning of the CERN Closed Loop gas system used by the RPC muon detectors in experiment CMS at the LHC. Studies include a three-phase campaign both in low-radiation (cosmic rays at the CERN ISR test site) and high-radiation (at the CERN Gamma Irradiation Facility) environment. We describe dedicated RPC chambers tests and chemical analysis of filters and gas used which will allow us to fully characterize the Closed Loop system in view of the upcoming commissioning of the CMS RPC detector.

1 INTRODUCTION

Resistive Plate Counters (RPC) are used in the muon detector of CMS at the CERN LHC. Gas mix used is typically 96% of $C_2H_2F_4$, 3.5% of iC_4H_{10} and 0.5% SF_6. Gas mix is humidified at the 45% RH level typically. Because of the high cost of freon and the very large gas volumes involved in the full system, the design of the CMS RPC gas system is based on a recirculation system called the Closed Loop (CL) system.

The absence of gas contaminants is of paramount importance in all gas detectors, and expecially in RPC due to the high reactivity of F-based gas mix used[1]. A gas gain monitoring system[2] has been designed and is now in construction by the CMS RPC Frascati group for the fast and accurate monitoring of the working point, based on monitoring of the avalanche charge and of the chamber efficiency. The gas gain monitoring system is based on three single-gap RPC subdetectors flushed with clean mix, CL mix after purifiers before crossing CMS RPC, and CL mix from the return lines. The gas gain monitoring will provide a warning in case of a working point change.

A gas quality monitoring system is being developed by the CMS RPC Bari group[3] and it will perform chemical (GC, HPLC, pH, etc) analyses following the warning condition released.

2 THE CMS CLOSED LOOP GAS SYSTEM

The CMS Closed Loop (CL) [3.1] gas system is shown in Fig.1. Tests with a scaled-down prototype CL system were performed in 2001 at the Gamma Irradiation Facility (GIF) [4]. In the CL system, gas purity is guaranteed by a multistage purifier system. Purifier filters were selected such that contaminants components (appearing as spurious peaks in a GC other than the mixture known gases) were minimized, i.e.: zeolite molecular sieve [11], BASF CuO-ZnO catalyst R3-12, BASF CuO R3 11G, Leuna $NiAlO_3$ catalyst. The absorption capacity for the various contaminants was not studied [5]. The prototype CL system is presently used at the ISR test site for RPC prior to installation in CMS. While the fraction of new gas planned for use at the full CMS RPC detector is 2%, the new gas fraction used at ISR is 10%.

The prototype CL system at ISR has been intensively used since the beginning of 2006. It was immediately noted how water saturation of filters occurred after about twenty days, followed after a few days by currents increase in the chambers (Fig. 2). Such a behavior is consistent by the presence of one or more unknown contaminants which are removed by purifiers as long as not saturated with water. Replacing and regenerating filters after about 25 days keeps the CL system up and running. Design of upgraded purifiers batteries [6] for use in CMS RPC is completed.
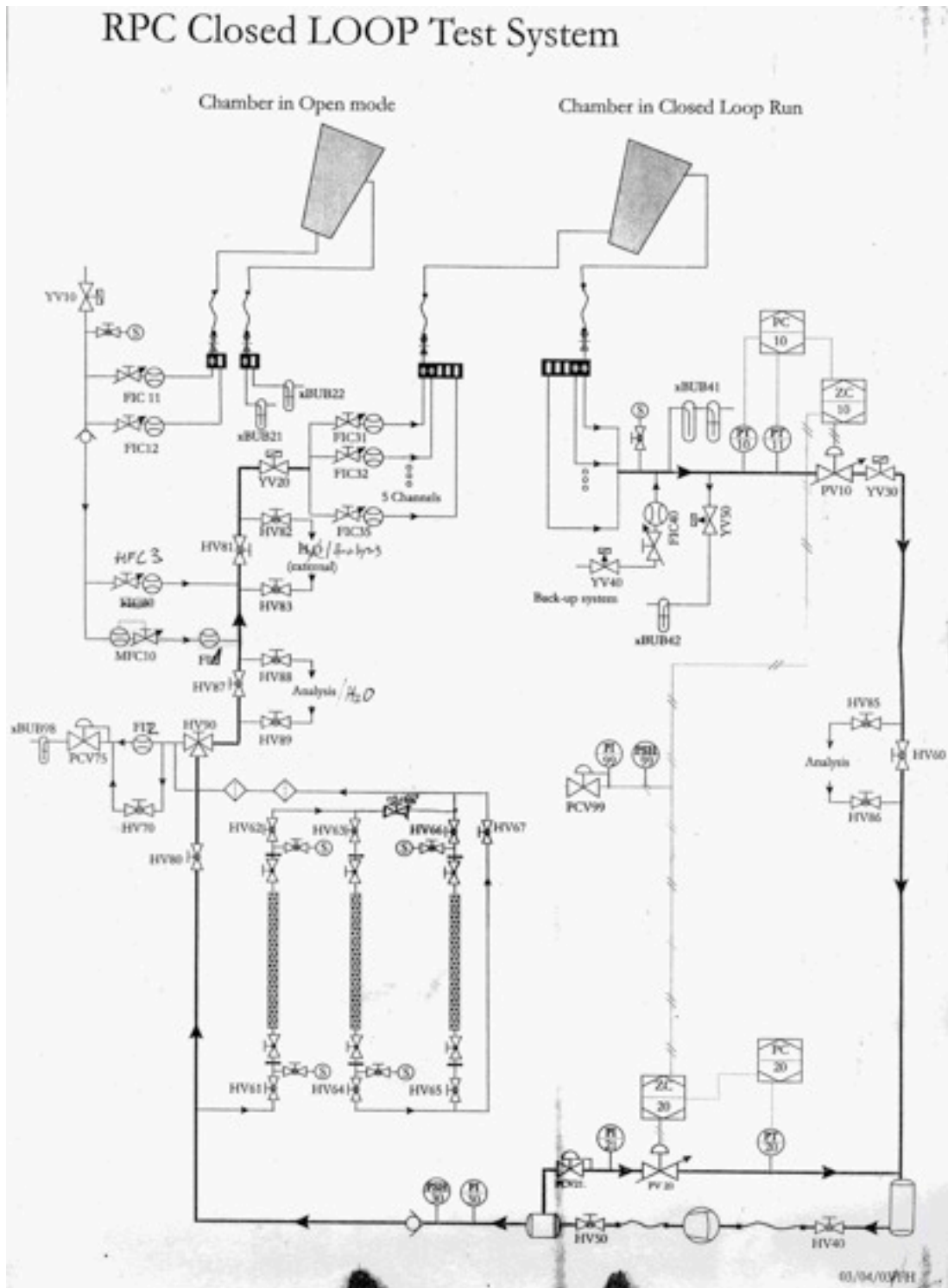
RPC Closed LOOP Test System

Figura 1 The prototype CERN CL system used at GIF and ISR for CMS RPC(Ref. [3.1]).
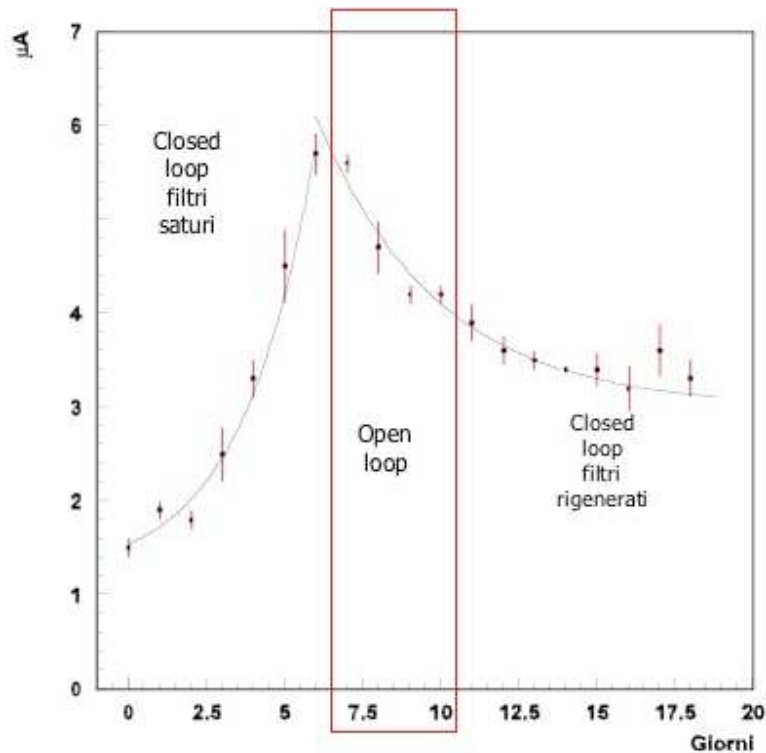
Figure 2 Typical current increase observed at ISR in CL when filters are saturated. Filters regeneration restores standard currents.

3 A THREE-PHASE ANALYSIS CAMPAIGN

No aspect of the chemistry of purifiers is fully understood, nor the extent or the nature of contaminants. Tests[7][8][9] at GIF showed the presence of HF, which is detected by accumulation methods or by the use of HPLC, or ionic column – equipped GC. We propose a systematic investigation aimed to fully clarify the chemistry of purifiers used in the CL. Our proposal is structured in three phases:

- PHASE 1 – Started on September 1st 2006 and currently in progress, we study the purifiers and gas in the CL system at the ISR during chamber testing. Priority is given to optimal and efficient completion of chambers prior to installation. No chambers stress is therefore possible, and currents have to be kept at their minimum. We take samples of unused, contaminated, and regenerated filter material, and perform SEM/EDS, diffractometric, and chemical analyses (see Sect. 4). We plan to understand the basic features of CL purification chemistry in a low-radiation environment.

- PHASE 2 – At completion of ISR chamber testing, possibly starting at beginning of 2007, we shall install new single gaps dedicated to contaminants studies. We shall allow high doses of contaminants to be produced and shall let currents increase. This will confirm the early indications and reproduce the contaminant effect observed in PHASE 1.

- PHASE 3 – High-radiation environment and contaminants will be studied at the CL at the GIF facility. We are considering reusing the setup utilized in 2001, possibly in collaboration with the ATLAS RPC group [10].

4. ON THE CHEMICAL STUDIES TO CHARACTERIZE MATERIALS AND COMPONENTS USED IN THE CL GAS SYSTEM

The understanding of both presence and extent of contaminants in the CL system calls for a detailed chemical analysis of filters, gas and materials used. We plan to perform measurements using

- SEM-EDS (Scanning Electron Microscopy- Energy Dispersive X-ray analyzer). SEM is a microbeam instrument that is used also in applied material science to analyze polished and/ or thin sections, as well as unmounted pieces of material. The SEM is used to identify minerals, to obtain photomicrographs that show the size and relationship of mineral grains, distribution of defects and to obtain X-ray images that can show the distribution of the elements and inclusions in minerals, compounds and materials. These analyses will be performed at Dep. of Ingegneria dei Materiali e delle Materie Prime (ICMMPM), University of Rome "La Sapienza", using a SEM Hitachi S2500, equipped with a Kevex X-ray Microanalizer.

- XRD (X-ray diffractometry) Every crystalline material has a X-ray diffraction (XRD) pattern that is dependent upon the crystal structure, and upon the composition of mineral. XRD pattern are obtained by X-ray diffraction, and are used to identify the compounds. In the case of the molecular sieve, XRD analyses permit us to identify the zeolitic filter used, and also to verify if contaminants have a crystal structure. These analyses will be performed at Dep. ICMMPM University of Rome "La Sapienza" using a Philips X'Pert Plus diffraction system, Copper Tube.

- LC /MS (liquid chromatography -mass spectrometry). Chromatographic analysis relies on the fact that when different substances are absorbed, suspended or dissolved in a medium, they travel through that medium at different rates, so that they get separated. It is possible to analyze anions in water solutions. These analyses will be performed at Dep. ICMMPM University of Rome "La Sapienza" using a HPLC ionic chromatography (High Performance Liquid Chromatography) composed by a Shimadzu LC-10ATvp pump and a column Shodex ICSI-50 4E.and a conductivity detector CDD-10Asp.

- GC/MS (gas chromatography -mass spectrometry) is useful for separating/analyzing mixtures of volatile, thermally stable organic compounds. GC separates organic compounds in a mixture based on differences in their partitioning behavior between a mobile gas phase (usually inert gases like He, Ar, N) and a stationary phase (usually liquid on a solid support) in a column. It is possible to analyze the gas/water mixture before and after the filters in order to evaluate the depletion in anions. At Dep. ICMMPM University of Rome "La Sapienza" we could use a gas chromatograph Varian 3300 with a FID detector and a Altech EC-20, AT 20 capillary column that can perform analyses for alcohol till C5 and for acetone. It is necessary to improve the instrument with a different column for halogen organic compound and a detector that can be a problem because of its radioactivity requiring peculiar safety measures.

- ICP-MS(Inductive Coupled Plasma-Mass Spectrometry). A multielement acid attack

dissolves the sample. The near total digestion is obtained employing HF, HClO4, HNO3 and HCl to get as much of the sample into solution as possible without fusing the sample, the resulting metals can be determined by ICP-OES (Inductive Coupled Plasma-Emission Spectroscopy). The partial extraction is analyzed by ICP-MS to provide lower detection limits. This analysis provides a trace elements scan for virtually all types of mineralization but also useful information on alteration, presence of contaminants in minerals, materials, compounds. In ICP-MS analyses detection limit varies from 0.01% for some elements to 0.2 ppb for others). The combined INAA, 4 acid digestion ICP and ICP-MS provide the most comprehensive near total identification using an acid digestion.

- XRF (X-ray fluorescence) major oxide determinations are performed by the fusion of the material by this old standard analysis (detection limit 0.01%). These analyses will be performed by ACTLABS Laboratories that achieved the ultimate accreditation to international standards, the ISO 17025 for specific registered tests, and CAN-P-1579 specific to mineral analysis laboratories.

Table 1 Filters chemical analyses.

SAMPLE	ANALYSES	
MOL.SIEVE 5A UNUSED		‖‖‖‖‖‖‖‖
MOL.SIEVE 3A UNUSED		‖‖‖‖‖‖‖‖
Ni Al$_2$O$_3$ UNUSED		
Cu CuZn UNUSED		
Cu PIPE UNUSED		▓▓▓▓▓▓
BAKELITE A OILED UNUSED		▓▓▓▓▓▓
BAKELITE B OILED UNUSED		▓▓▓▓▓▓
BAKELITE C OILED GRAPHITE UNUSED		
MOL.SIEVE 5A USED		‖‖‖‖‖‖‖‖
Ni Al$_2$O$_3$ USED		
Cu CuZn USED		
BAKELITE A OILED GRAPHITE USED		▓▓▓▓▓▓
BAKELITE B OILED GRAPHITE USED DEFECT		▓▓▓▓▓▓
MOL.SIEVE 5A REGENERATED		‖‖‖‖‖‖‖‖
MOL.SIEVE 3A REGENERATED		‖‖‖‖‖‖‖‖
Ni Al2O3 REGENERATED		
Cu CuZn REGENERATED		
WHITE SAMPLE - NATURAL ZEOLYTE		‖‖‖‖‖‖‖‖
LEGENDA		
X-Ray Fluorescence		
ICP (Inductive Coupled Plasma-Mass)		
INAA (Instrumental Neutron Activation Analysis)		
ICP-MS (Inductive Coupled Plasma-Mass Spectrometry)		
XRF (Total Identity +S, F, C organic, C total, C graphic)		
XRD (X-ray Diffraction)		
SEM-EDS (Scan. Electron Micr. - Energy Dispersive Spectrom.)		

We plan to perform cyclic sampling. A typical filter sampling cycle study will be made of samples taken in various phases of gas cycle, i.e.

1. take a fraction (20-50 g) of the new molecular sieve just before inserting it in the gas system;

2. take a fraction (20-50 g) of the used molecular sieve just before regeneration;
3. take a fraction (20-50 g) of the regenerated molecular sieve;
4. sample gas just before the filtering;
5. sample fresh gas.

Same procedure will be followed for Ni-Al$_2$O$_3$, Cu-CuZn. A complete list of chemical analyses aimed to fully characterize the filters used, and to ascertain both presence and nature of contaminants is shown in Tab.1.

The sampling of gas mixture is a complex operation which requires hardware implementations to the CL system. The CL system works at virtually zero pressure, adequate pumping and switching has to be designed and installed for gas sampling. Analysis of gas samples will be performed mainly at CERN in collaboration with the CERN Gas Group.

6. PHASE 1: ANALYSIS CAMPAIGN AT THE CERN ISR CLOSED LOOP SYSTEM DURING TEST OF CMS RPC CHAMBERS

Samples of purifiers were taken starting on September 1st, 2006. The molecular sieve [11][12][13] was found a mixture of 5A sample with a grain size in the range of 2.0 mm to 2.8 mm, and a 3A sample with a grain size in the range of 1.0 mm to 1.4 mm. Chemistry of 3A, 4A and 5A is different. The best known synthetic zeolites are Linde Type A (LTA). The LTA 4A type is a sodium form of zeolite, with a pore opening of approximately 4Å, called 4A molecular sieve. If the sodium ion is exchanged with the larger potassium ion, the pore opening is reduced to approximately 3Å (3A molecular sieve). On the other hand, when calcium is exchanged, one calcium ion replaces two sodium ions: thus, the pore opening increases to approximately 5Å (5A molecular sieve). Ion exchange with other cations is sometimes used for particular separation purposes. 5A zeolites were chosen during tests in 2001 at GIF.

The first analyses (XRD) and granulometric size distribution have been performed on
1) an unused 5A sample (grain size in the range of 2 mm to 2.8 mm was measured), with XRD results in fig. 2;
2) a used 5A sample (grain size in the range of 1 mm to 1.4 mm was measured);
3) an unused 3A sample (grain size in the range of 1 mm to 1.4 mm was measured).

Preliminary results on XRD and granulometry showed that the filters in use were a mixture of 3A and 5A. Following these findings, filters were opened, and filter material completely replaced with unused material. The filters composition is now:
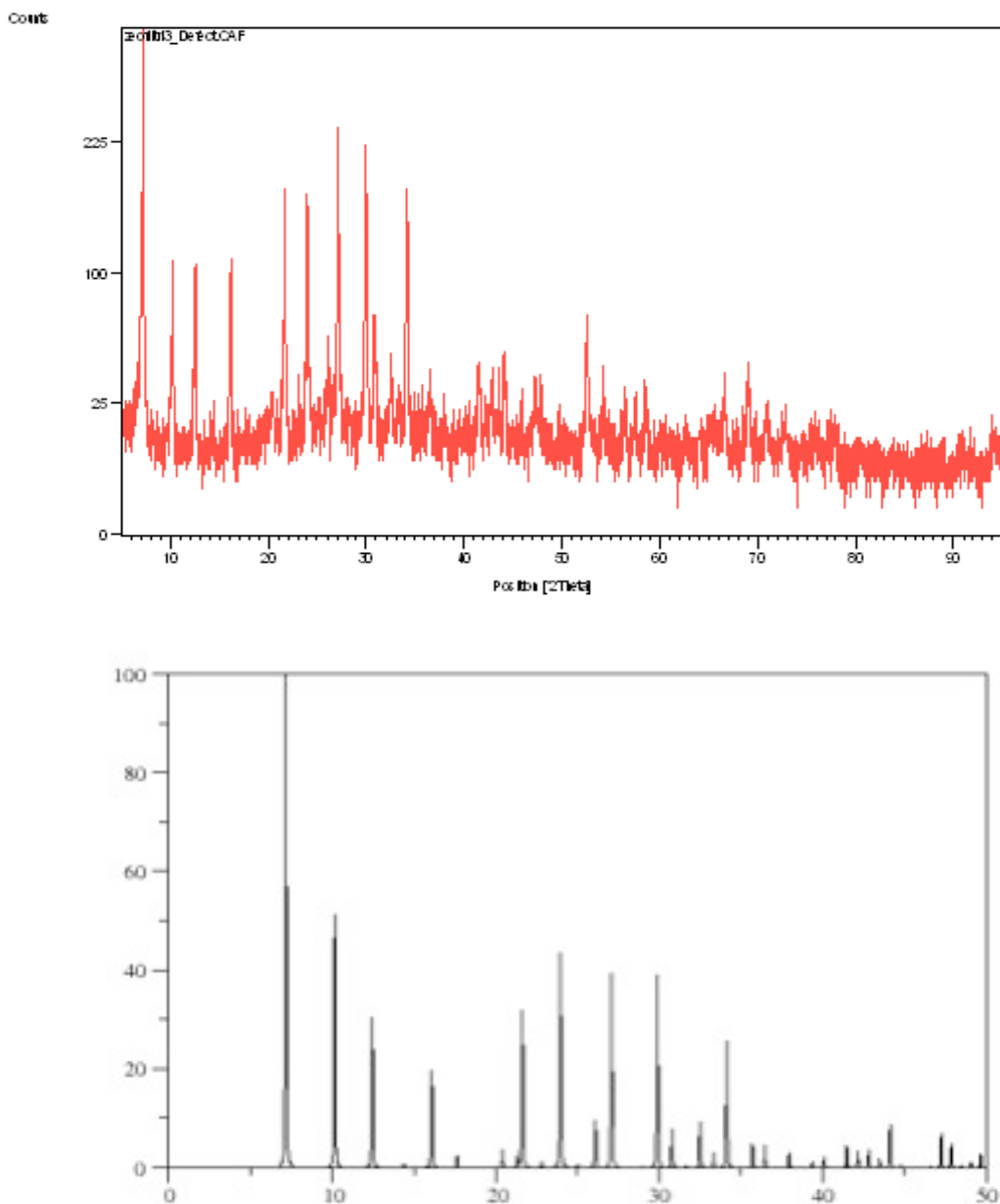
Figure 2 XRD spectrum a) unused filter material from ZEOCHEM zeolite 5A as analyzed at Univ. Rome; b) standard zeolite Type A by LINDE[12]. Peaks positions well match and confirm chemical structure.

- Molecular sieve 5A;
- Metallic filter $Ni-Al_2O_3$;
- Metallic filter composed of 50% R3-11/G and 50% R3-12. R3-11/G filter is CuO, while R3-12/G is made of CuO and ZnO.

Both metallic filters release a very fine dust residue. We checked on the technical specs and the presence of graphite is confirmed. Such a discovery calls for a particulate filter to be installed downstream the purifier.

Filter must be regenerated even when unused. The molecular sieve is dried up by heating, while the metallic filters are regenerated by high-temperature flushing with a Ar/H_2 mix.

Hydrogen reduces CuO into Cu. In such a way the filter stops oxygen by oxidizing.

We performed a XRD of the unused 5A molecular sieve material (Fig. 3a) and compared it with existing data (Fig. 3b). The unknown compound well matches zeolite Linde Type A.

5 CONCLUSIONS

We have described the studies performed so far on the CL gas system of the RPC muon detectors in experiment CMS. Monitoring the gas quality is of paramount importance. We propose a systematic measurement campaign aimed to fully characterize the chemistry of purifiers used in CL, and to determine both nature and amount of unknown contaminants produced during RPC operation with F-based gas mix. Such a campaign has started already during RPC chamber testing at ISR, and should be continued by dedicated use of ISR setup at chamber test completion, and at GIF for studies in high-radiation environment.

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