A study of gas contaminants and interaction with materials in RPC closed loop systems

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A study of gas contaminants and interaction with materials in RPC closed loop systems


ABSTRACT: Resistive Plate Counters (RPC) detectors at the Large Hadron Collider (LHC) experiments use gas recirculation systems to cope with large gas mixture volumes and costs. In this paper a long-term systematic study about gas purifiers, gas contaminants and detector performance is discussed. The study aims at measuring the lifetime of purifiers with new and used cartridge material along with contaminants release in the gas system. During the data-taking the response of several RPC double-gap detectors was monitored in order to characterize the correlation between dark currents, filter status and gas contaminants.

KEYWORDS: Muon spectrometers; Resistive-plate chambers

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1 Introduction

Resistive Plate Counters [1] (RPC) detectors are installed at both the ATLAS (A Toroidal LHC Apparatus) [2] and CMS (Compact Muon Solenoid) [3] experiments at the LHC (Large Hadron Collider) of CERN, Geneva (Switzerland) to provide triggering and synchronization in both barrel and endcap regions as part of the muon system. RPCs use a freon-based gas mixture (typically 95.2% C₂H₂F₄ - 4.5% Iso-C₄H₁₀ - 0.3% SF₆) in a recirculation system call Closed Loop (CL) [4]. Gas mixture and ambient are humidified at the 40% RH level for two main reasons. The first is to keep the gap resistivity at a constant value. The second reason concerns the fact that an unbalance RH level would result in a mechanical stress of the phenolic-melaminic laminate [5] given its high hygroscopy. The CL was designed to cope with large gas mixture volumes and costs. In the closed loop system industrial filters commercially available are in operation to purify the mixture and to prevent contamination collection that affects the RPC performances.

A systematic study of CL gas purifiers has been carried out from 2008 to 2011 at CERN using RPC chambers exposed to cosmic rays and operated with a downscaled version of the closed loop gas system used for CMS, equipped with several sampling points for gas analysis. Goals of the study [6] were to observe the release of contaminants in correlation with the dark current increase in RPC detectors and to measure the purifier lifetime [7] with new material. In this paper, new preliminary results from the 2011 run are shown new preliminary results characterizing the purifiers in use and study the pattern of dark currents increase in the upstream versus the downstream gaps.

2 Experimental setup

The experimental setup [8, 9] is composed of a closed and an open loop gas systems (figure 1). To purify the closed loop gas mixture, commercial filters are used as shown in figure 2. The gas is recycled at 90%, thus 10% of the gas volume is supplied at each cycle from fresh gas. The gas volume change rate, for each gap, is $\approx 1$ vol/h.
The first purifier acts as a molecular sieve, the cartridge is filled with two different zeolites (ZEOCHEM [10]) 5Å (10%) and 3Å (90%). The second purifier cartridge is filled with 50% Cu-Zn filter type R12 (BASF [11]) and 50% Cu filter type R3-11G (BASF) while the third purifier consists of NiAlO$_3$ filter type 6525 (LEUNA [12]).
Eleven double-gap RPC detectors are installed in a confined space (hut) with controlled and monitored temperature and humidity. Figure 3 and figure 4 show that temperature and RH are kept constant at 20 ± 1° C and 40 ± 5% RH.

Nine detectors out of eleven are operated in CL mode while two are operated in open loop (OL) mode. Each RPC detector has two gaps connected in series and defined as upstream and downstream whose gas lines are serially connected. The detectors are operated at a 9.2 kV applied voltage supply, the CMS chosen working point [13] for long term studies. At the working point selected, the anode dark current drawn, read by the power supply, is approximately 1–2 µA [13] (figure 5).

The humidity of the open and closed loop gas mixture are monitored (before and after the gap) and controlled to be constant at 40% level. Two independent dedicated gas humidifiers control the water content of the gas mixture in order to run with a constant value. Given the humidity equilibrium between gas mixture and ambient and the online monitoring of amount of water inside the gas the RPC under test were operated without any release or absorption of water. Gas sampling points, before and after each filter of the closed loop, allow chemical and gas chromatograph (GC) analysis [8]. Gas mixture composition is monitored twice a day by GC, which also provides the amount of air contamination, stable over the entire data-taking run and below 300 (100) ppm in closed (open) loop (figure 6) thanks to the purifiers installed in the closed loop system.
3 Chemical analysis setup

Chemical analyses have been performed in order to correlate the increase of dark currents with the release of gas contaminants. To identify the contaminants nature, the gas is sampled before and after each purifier, and bubbled into a set of PVC (Polyvinyl chloride) flasks (figure 7) through teflon filters that act as particulate sieves.

The set of PVC flasks consists in four serially connected flasks where the gas (1 mbar over-pressure each flask), sampled from the closed loop system, is flushing. Flask 1 acts as a buffer to avoid return of LiOH into the CL. Flasks 2, 3 and 4 contained 250 ml solution of LiOH (0.001 mol/l corresponding to 0.024 g/l, optimised to keep the pH of the solution at 11). After flask 4, the gas mixture is sent to the exhaust. The bubbling of gas mixture into the flasks 2, 3 and 4 allows one to capture a wide range of elements that are likely to be released by the system, such as Ca, Na, K, Cu, Zn, Ni, F. These elements are collected in the flasks and revealed, at the end of the data-taking, when the LiOH solution is being analyzed with ionic chromatography. Moreover the flow of each sampling is measured to estimate the total amount of gas for the whole period of data-
Figure 7. Chemical analyses are performed using LiOH flasks in which gas is bubbled and contaminants collected.

Figure 8. Calibration curves of fluoride monitoring sensors.

taking. Sampling points HV61 and HV64 (figure 7) are located before filters (HV61), after zeolite filter (HV62), after Cu/Zn filter (HV64), after Ni filter (HV66).

The fluoride production of RPCs in CL was measured previously in high-radiation condition [16, 17]. To measure the fluoride production, sampling point HV61 and HV62 are equipped with two additional flasks and fluoride selective electrodes. The F$^-$ selective electrode adopted [14] is a solid state half-cell sensor that requires, as a separate reference, a silver-silver chloride double junction half-cell reference electrode. Selective electrodes are installed to measure the ionic potential, which is directly connected to the ionic concentration. The selective electrodes monitor the collected amount of ions integrated over time by means of a custom software which logs the electrochemical potential every 10 minutes in order to estimate the F$^-$ production rate and concentration in the system. Both electrodes (reference and sensor) are immersed in a diluted TISAB II solution [15]. This increases and stabilizes the ionic strength of the solution making a linear correlation between the logarithm of the concentration of analyte and the measured potential. The selective electrodes were calibrated at the beginning of the run and also during the run itself to double-check a possible shift of the factory settings. Standard solutions containing 0.001 mg/l, 0.005 mg/l, 0.01 mg/l, 0.10 mg/l, 1.0 mg/l, 10.0 mg/l, 20.0 mg/l, 50.0 mg/l concentration of F$^-$ are used for absolute calibration. Figure 8 shows the calibration curves.
Table 1. Summary table of Closed Loop (CL) and Open Loop (OL) channels.

<table>
<thead>
<tr>
<th>Run</th>
<th>Cycle</th>
<th>Period</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>29/08/2008 - 11/10/2008</td>
<td>stable currents CL new filters, 9 ch CL, 2 ch OL</td>
</tr>
<tr>
<td>1</td>
<td>2</td>
<td>12/10/2008 - 22/01/2009</td>
<td>stable currents CL new filters 9 ch CL, 2 ch OL</td>
</tr>
<tr>
<td>1</td>
<td>3</td>
<td>23/01/2009 - 28/04/2009</td>
<td>increasing currents CL new filters 9 ch CL, 2 ch OL</td>
</tr>
<tr>
<td>1</td>
<td>4</td>
<td>29/04/2009 - 14/07/2009</td>
<td>increasing currents CL new filters 9 ch CL, 2 ch OL</td>
</tr>
<tr>
<td>1</td>
<td>5</td>
<td>15/07/2009 - 27/07/2010</td>
<td>decreasing current OL used filters 0 ch CL, 11 ch OL</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>28/07/2010 - 07/01/2011</td>
<td>stable currents CL used filters 7 ch CL, 2 ch OL</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>08/01/2011 - 05/07/2011</td>
<td>increasing currents CL used filters 7 ch CL, 2 ch OL</td>
</tr>
</tbody>
</table>

**Figure 9.** Upstream gap total current during all runs with $F^−$ production.

### 4 Results and discussion

To describe the operating conditions (table 1) the data-taking period is divided into two runs over three years. During the whole data-taking we operated the system with cosmic rays to start studying and clarifying several issues such as contaminants identification, HF production rate and purifiers lifetime.

Each run is characterized by cycles of operation. Figure 9 shows the average of all RPC anodic dark currents $I_i(t)$ over $n$ gaps, normalized by their initial values $I_i(t_0)$, each gap is read out separately. The $z$-axis scale (color-coded) shows the $F^−$ produced by the system. The increase of dark anode currents of up-stream gaps in run 1 and run 2 is clearly visible, as well as the increase of $F^−$ concentration. The dark currents of down-stream gaps, as well as the currents of all RPCs in OL, are found stable.

Before run 1 few weeks tests were performed without any RPC in the closed loop and with
Figure 10. Dark currents increase (run 1) in the up-stream gap and not in the down-stream gap, correlated to the detection of gas contaminants measured using the chemical analysis setup.

RPC turned off, as reference measurement for chemical analyses. The release of contaminants was found negligible and within the experimental errors. In run 1, the purifier cartridges are filled with new material. Eleven double-gap RPC detectors are used, nine in CL and two in OL mode. Cycle 1 and cycle 2 have stable currents up to April 2009, when an increase in the dark current occurs for all up-stream gaps in CL, leaving the down-stream gaps stable. Cycle 4 in particular shows a clear increase of currents, and was terminated before permanent damage occurred to the detector. The lifetime of purifiers is determined by evaluating the duration of cycle 1 and cycle 2

$$\tau_{\text{run 1}} = 211 \pm 2 \text{ days}$$

The total gas flow is $63 \pm 3$ l/h. We measure the fluoride production (figure 11) during run 1 as $1.10 \pm 0.05 \mu\text{mol}/\text{l}$ corresponding to a total accumulation in the CL of $(45 \pm 2) \times 10^3 \mu\text{mol}/\text{l}$. The purifier lifetime normalized to the F$^-$ production is

$$\hat{\tau}_{\text{run 1}} = 4.64 \pm 0.24 \text{ days/mmol/l}$$

Figure 10 shows the typical behaviour of one RPC detector in closed loop correlated with the concentration of the main contaminants found.

Before starting run 2, all purifiers are regenerated following the CERN gas group standard procedure, i.e. by means of a flushing of hot ($215^\circ\text{C}$) Ar and H$_2$ mixture (80:20) for twelve hours. In this run nine double-gap RPC detectors are used (seven in CL and two in OL mode) since two double-gap did not recover their initial low currents. The average gas volume change rate is $\approx 1 \text{ vol/h}$ while the total measured flux is $\approx 54 \pm 31$ l/h. The currents of down-stream gaps are found stable throughout all cycles as in run 1, while the currents of the up-stream gaps increase. As a cross-check, gas supplies of two gaps of the same RPC detector were swapped to check that in a pair of gaps only the up-stream gap showed currents increase. The lifetime of regenerated purifiers is evaluated:

$$\tau_{\text{run 2}} = 160 \pm 2 \text{ days}$$
Figure 11. $F^-$ production during run 1 (new filters) and run 2 (used filters).

The $F^-$ production is measured $0.84 \pm 0.05 \mu\text{mol}/l$ (figure 11), corresponding to an accumulation of $33 \pm 2 \times 10^3 \mu\text{mol}/l$. The purifier lifetime normalized to the $F^-$ production is

$$\hat{\tau}_{\text{run 2}} = 4.68 \pm 0.25 \text{ days/mmol/l}$$

(4.4)

Analyses are in progress in order to confirm the release of contaminants observed in run 1 and shown in figure 10.

The lesser $F^-$ production in run 2 with respect to run 1 is interpreted as due to the smaller number of detectors used in run 2. Although the lifetime of purifiers is measured different in run 1 and run 2, the lifetime normalized to the $F^-$ production is found compatible within errors, i.e., $4.64 \pm 0.24 \text{ days/mmol/l}$ for run 1 and $4.68 \pm 0.25 \text{ days/mmol/l}$ for run 2.

During both run 1 and run 2 the zeolite filter removes effectively part of the produced $F^-$, in particular in the last run filters seem to perform slightly better, as expected, since the filtering performance improves after few regeneration processes. The presence of an excess production of K and Ca in coincidence with the currents increase also suggests a damaging effect of HF \cite{17}, produced in the system, on the K- and Ca-based zeolite framework. Further analyses are ongoing to verify the presence of contaminants in run 2.

5 Conclusions

Preliminary results on studies of contaminants, and on characterizations of materials and gas used in the CL gas system of the CMS RPC muon detector were reported. Quantitative gas chemical analysis were performed by using GC, pH sensors and contaminants detectors. The lifetime of new purifiers is compatible with the lifetime of regenerated purifiers when normalized to the $F^-$ produced in the system. The anodic dark current of up-stream gaps increases when purifiers are
exhausted, while the down-stream gaps show stable current. This behavior is suggestive of a mechanical filtering, i.e., the first gap acts as a filter to the second gap which does not receive a polluted gas mixture. Finally very preliminary results from run 1 seem to suggest a correlation between the increase of dark current and the release of K and Ca but analysis of run 2 are in progress to confirm this effect. Further studies are in progress in order to ascertain the presence of such contaminants in run 2 with regenerated purifiers.

References


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