

AN ANALYSIS OF MATERIALS USED IN THE RPC DETECTOR AND IN THE CLOSED LOOP GAS SYSTEM OF CMS AT THE LHC

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Abstract

The results are reported of the study of materials used in the CERN Closed Loop recirculation gas system currently under test with the RPC muon detectors in the CMS experiment at the LHC. Studies include a sampling campaign in a low-radiation environment (cosmic rays at the CERN ISR test site). We describe the dedicated RPC chamber tests, the chemical analysis of the filters and gas used, and discuss the results of the Closed Loop system.

1 Introduction

Resistive Plate Counters (RPC) are used in the muon detector of the Compact Muon Solenoid (CMS) experiment at the CERN Large Hadron Collider. RPC use fluorine-based gas mix whose main component is Freon. 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 especially in RPC due to the high reactivity of the F-based gas mix used [2] [3]. A gas gain monitoring system [4] has been designed, built and is being commissioned by the CMS RPC group for the fast and accurate monitoring of the working point, based on the 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 group which will perform chemical analyses such as Gas Chromatography (GC), High Pressure Liquid Chromatography (HPLC), pH meters, etc. following the release of a warning condition.

No aspect of the chemistry of purifiers used in CL is fully understood, nor the extent or the nature of contaminants with the exception of fluoridric acid, whose presence is established. Tests [7],[8] at the Gamma Irradiation Facility (GIF) [9] showed the presence of HF, which is detected by accumulation methods or by the use of HPLC, or ionic column-equipped GC. We have proposed [11] a systematic investigation aimed to fully clarify the chemistry of purifiers used in the CL, structured in three phases. Purifiers and filters were firstly studied at the ISR test area during chamber testing with cosmic rays (phase one). At completion of ISR chamber testing, new RPC detectors dedicated to contaminant studies will be deployed (phase two). The system behavior will be studied by allowing currents to increase under production of contaminants. Finally, high-radiation environment and contaminants will be studied at the CL at the GIF facility (phase three). Results reported on in this paper refer to phase one which took place in September 2006.

2 The Closed Loop system of the CMS RPC detector

The CMS Closed Loop (CL) [10] gas system is shown in Fig. 1. Tests with a scaled-down prototype CL system were performed in 2001 at the GIF [7][8][12]. 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

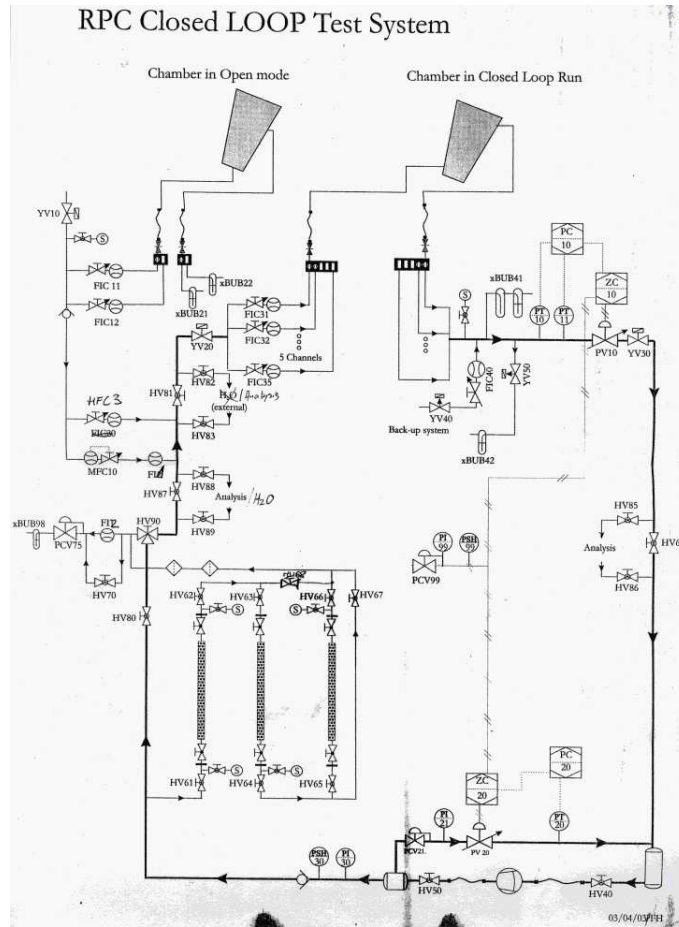


Figure 1: Closed Loop (from ref [10])

mixture known gases) were minimized, i.e.: zeolite molecular sieve[14], BASF CuO-ZnO catalyst (R3-12) [15], BASF CuO (R3-11G)[15], LEUNA NiAlO₃ catalyst[16]. The absorption capacity for the various contaminants was not studied[12]. The prototype CL system was used at the ISR test site for RPC prior to installation in CMS. While the fraction of fresh gas planned for use at the full CMS RPC detector is 2%, the fresh gas mix 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 that water saturation of filters occurred after about twenty days, followed after a few days by an increase of currents in the chambers (Fig. 2). In the ISR setup, two RPC gaps are connected in series. Currents of upstream gap are observed rising much more than currents in the downstream gap. Such a behavior is consistent with the presence of one or more unknown contaminants generated by both gaps, that

accumulate in the upstream gap which acts as a filter. Contaminants 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.

3 Filters

With the original selection criteria being the filtering of H₂O and O₂, tests at GIF showed GC peaks of unknown pollutants that disappeared after the following purifiers:

- purifier 1. consisting of a 24-litre cartridge filled with 5Å Type molecular sieve manufactured by ZEOCHEM
- purifier 2: consisting of two 24-litre cartridges, each filled with a combination of:
 1. 25% Cu-Zn filter type R12 manufactured by BASF
 2. 25% Cu filter type R3-11G manufactured by BASF
 3. 50% Ni AlO₃ filter type 6525 manufactured LEUNA

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. 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.

Replacement and regeneration of filters after about 25 days keeps the CL system up and running. The regeneration procedure followed consists of heating at 240°C in a H₂/Ar (7/93) flux of 100 liters/hour for 16 hours, followed by a 2-hour cleaning with Ar. Design of upgraded purifiers cartridge batteries [13] for use in CMS RPC is completed. In the following sections we discuss the filters currently used.

3.1 Molecular sieve 5Å Type by ZEOCHEM

Zeolite molecular sieves are crystalline, highly porous materials, which belong to the class of aluminosilicates. These crystals are characterized by a three-dimensional pore system, with pores of a precisely defined diameter. The corresponding crystallographic structure is formed by tetrahedrons of (AlO₄) and (SiO₄). These tetrahedrons are the basic building blocks for various zeolite structures, such as zeolites A and X, the most common commercial adsorbents.

Due to the presence of alumina, zeolites exhibit a negatively charged framework, which is counter-balanced by positive cations resulting in a strong electrostatic field on

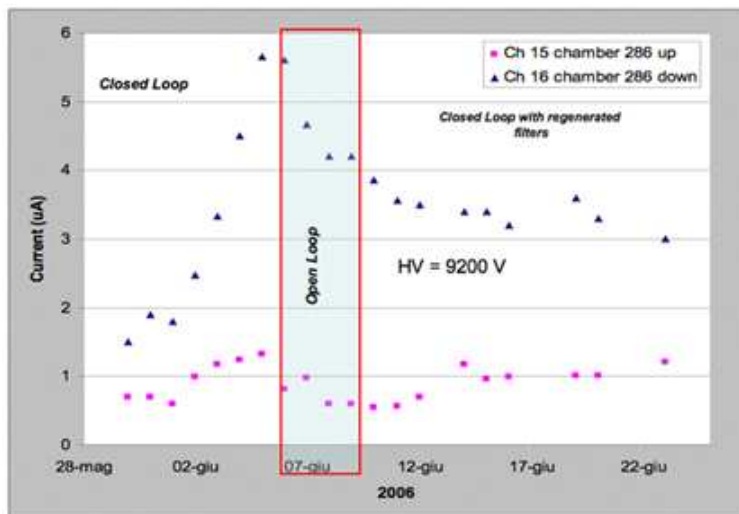
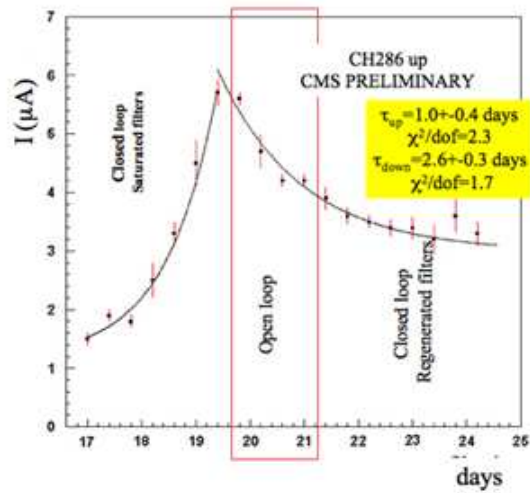
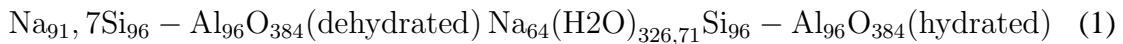


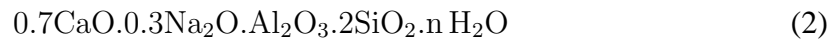
Figure 2: Increase of currents in RPC chambers under test at the ISR in CL gas system when purifiers are saturated. Currents start decreasing when chamber is set in Open Loop, and decrease further when chamber is returned to CL with regenerated purifiers. Top inset show results of a best fit of an exponential curve to data.

the internal surface. These cations can be exchanged to fine-tune the pore size or the adsorption characteristics. For instance, the sodium form of zeolite type A has a pore opening of approximately 4Å, and it is called a 4Å molecular sieve. If the sodium ion is exchanged with the larger potassium ion, the pore opening is reduced to approximately 3Å (3Å molecular sieve). On ion exchange with calcium, one calcium ion replaces two sodium ions. Thus, the pore opening increases to approximately 5Å (5Å molecular sieve). Ion exchange with other cations is sometimes used for particular separation purposes [23].

Zeolite A, like other zeolites, is synthesized in a gelling process. Sources of alumina (usually sodium aluminate) and silica (usually sodium silicate) are mixed in basic water solution to give a gel. The alkali agent can be NaOH or solutions of quaternary ammonium salts, amines, or other polar organics. The gel is then heated to 70-300 °C to crystallize the zeolite. The zeolite is normally synthesized in the Na⁺ form. This can account for Na presence in the 5Å zeolite a Ca LINDE Type A, the non-complete ion exchange of Na⁺ by Ca⁺⁺ can justify a 5% of NaO in CL molecular sieve. Chemical composition of standard LINDE Type A zeolite is [18–22]



ZEOCHEM [14] Molecular Sieve type 5Å is an alkali alumino Silicate, and it is the calcium form of the Type A crystal structure. Typical chemical formula for ZEOCHEM molecular sieve 5Å is



Type 5Å has an effective pore opening of 5Å. It is especially suitable for Pressure Swing Adsorption (PSA) applications where it may be used for the separation of normal- and iso-paraffins (C4 to C6 species), in PSA hydrogen purification and in oxygen concentrators. It is an excellent adsorbent to remove water, CO₂, H₂S from sour natural gas streams, while minimizing COS formation. Light mercaptans are also adsorbed. It is also used for the separation of normal- and iso paraffins, and production of high purity N₂, O₂, H₂ and inert gases from mixed gas streams. ZEOCHEM molecular sieve Type 5Å can be regenerated by either heating in the case of thermal swing processes; or by lowering the pressure in the case of PSA processes. To remove moisture from a 5Å molecular sieve, a temperature of 250-300°C is required. A properly regenerated molecular sieve can give moisture dew points below -100 °C, or mercaptan or CO₂ levels below 2 ppm[14].

3.2 Cu-Zn filter type R3-12 by BASF (25%), Cu filter type R3-11G from BASF (25%).

BASF Catalyst R3-12[15] is designed for removal of arsine, phosphine, H₂S and COS in the treatment of process and product streams. Catalyst R3-12 is widely used in the purification of polymer grade propylene, and in guard beds to protect noble metal catalysts from arsenic and sulphur. Arsenic and sulphur react with the Cu and Zn constituents in the catalyst to form stable metal complexes which do not migrate or desorb off of the catalyst, therefore making R3-12 not regenerable. The composition is nominally 40 wt.% CuO, 40 wt.% ZnO, and 20 wt.% Al₂O₃.

BASF Catalyst R3-11G[15] is designed for regenerative removal of O, CO, H and others from industrial gases and liquids, composition is CuO. Capacity and lifetime vary, depending on levels of arsine and sulphur, on the space velocity, and other factors.

3.3 Ni-AlO₃ filter type 6525 by LEUNA (50%)

LEUNA Catalyst Ni-AlO₃ filter type 6525[16] is used for hydrogenation of benzene, phenols and cresols, reduction amination of alcohols, aldehydes and ketones into the corresponding amines, methanations. The catalyst consists of approximately 60 wt % Ni in alumina tablet form. The data sheet suggests the need to inquire the company for a suitable regeneration procedure. It is declared vulnerable to humidity, and the presence of very fine dusty particulates which calls for a particulate filter.

3.4 Filters sampling and analysis

Zeolites and other filters have been sampled (unused, contaminated, regenerated). Chemical analyses were performed on these samples for major and trace elements, as well as for sulphur and carbon (graphitic, total and organic), Ionic Chromatography (IC) to analyze fluorine, XRD analyses to identify zeolite type and to evaluate a possible crystalline structure of compounds, possibly occurring after gas filtering. The aims of these analyses are to characterize contaminants in filters, to identify their form (crystalline, in solution, amorphous), and to define the filters composition after regeneration (Tab.1).

The zeolite grain size and diffractometric pattern was studied. We found that the molecular sieve material consisted of a mixture of a 5Å type and a 3Å type zeolite, with the 5Å sample being measured with a grain size in the range of 2.0 mm to 2.8 mm, and the 3Å sample with a grain size in the range of 1.0 mm to 1.4 mm. Following these findings filters were opened, and filter material completely replaced with unused material. The composition of the filters used for the measurements reported in this paper is 100% molecular sieve 5Å type. We performed a XRD of the unused 5Å molecular

Table 1: Synopsis of chemical analyses performed on filters, gas and bakelite.

SAMPLE	ANALYSES
MOL. SIEVE 5Å UNUSED	1,2
MOL. SIEVE 3Å UNUSED	1,2
Ni Al ₂ O ₃ UNUSED	1
Cu CuZn UNUSED	1
Cu PIPE UNUSED	1,3
BAKELITE A OILED UNUSED	1,3
BAKELITE B OILED UNUSED	1,3
BAKELITE C OILED GRAPHITE UNUSED	1
MOL. SIEVE 5Å USED	1,2
Ni Al ₂ O ₃ USED	1
Cu CuZn USED	1
BAKELITE A OILED GRAPHITE USED	1,3
BAKELITE B OILED GRAPHITE USED DEFECT	1,3
MOL. SIEVE 5Å REGENERATED	1,2
MOL. SIEVE 3Å REGENERATED	1,2
Ni Al ₂ O ₃ REGENERATED	1
Cu CuZn REGENERATED	1
WHITE SAMPLE NATURAL ZEOLYTE	1,2
GAS BEFORE AND AFTER FILTERS	4
ABBREVIATIONS	
1	X-Ray Fluorescence ICP (Inductive Coupled Plasma) INAA (Instrumental Neutron Activation Analysis) ICP-MS (Inductive Coupled Plasma-Mass Spectrometry) XRF (Total Identity +S,F,C organic, C total, C graphitic)
2	XRD (X-Ray Diffraction)
3	SEM-EDS (Scan. Electron Micr. Energy Dispersive Spectrum)
4	HPLC (High Pressure Liquid Chromatography)

sieve material and compared it with existing data. The unknown compound well matches zeolite LINDE Type A.

An increase of trace elements in filters after operation and regeneration is observed. In zeolite molecular sieve, Cu, Ni and Zn increase after purification and only partially are removed after regeneration (Fig. 3), and this is also shown for the same for F and S (Fig. 4) and major elements (Fig. 5).

The zeolite molecular sieve filters not only H₂O, but also trace elements leached during the gas flux from the CL system. At this stage of purification, the zeolite acts as a buffer and pH of gas mixture increases from pH 4.9 to pH 6.0. Besides buffering, zeolite is attacked by HF formed in the gas mix, and the structure is deteriorated (further XRD analyses could verify the zeolite structure after filtration). This effect is confirmed by our chemical analyses which show a depletion in Al₂O₃ and SiO₂ that could signify a modification of structure from zeolitic to something else.

Analyses of LEUNA 6525 Ni-Al purifier show a decrease of Cr, Cu and Zn after purification, and only a partial recover of these elements after regeneration, i.e., Cr, Cu and Zn are released in the system and they are not completely recovered after regeneration

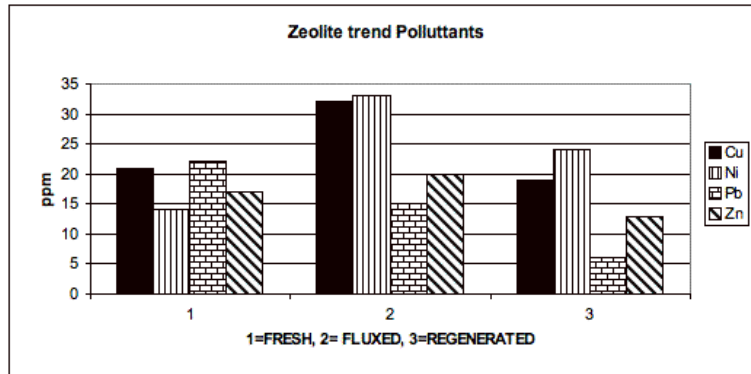


Figure 3: Zeolite trace elements trend (Cu, Ni, Pb, Zn)

(Fig. 6, 7). F and S increase after filtration in the LEUNA filter, but are not removed after regeneration (Fig. 8).

In the R3-12/R3-11G filter the F and S are trapped but not released after regeneration (Fig. 9). Substantial issues are raised by regenerating the R3-12 purifier, which is declared not regenerable in datasheet. R3-12 filter is usually used for removing traces of arsine, phosphine, COS, H₂S from vapor or liquid phases. Such components form CuS and ZnS sulphides which neither migrate nor desorb off of the catalyst. S is present in the gas mix as SF₆, and if released, S could bind with Cu and Zn in a sulphide form. This is confirmed by the Ni-based LEUNA purifier high S content, which remains in purifier even after regeneration.

Finally, both metallic filters release a very fine dust residue. We checked on the technical specs and the presence of graphite is confirmed. Particulate filters (20µm) should be installed.

4 Gas

Gas mix used is typically 96.2% of C₂H₂F₄ in its environmental-friendly version R137a, 3.5% of *i*C₄H₁₀, and 0.3% SF₆ to suppress streamer and operate in pure avalanche mode. Gas mix is humidified at the 45% RH level typically to balance ambient humidity, which affects the resistivity of highly hygroscopic bakelite, and to improve efficiency at lower operating voltage. The CL is operated with a fraction of fresh mix continuously injected into the system. Baseline fresh mix fraction for CMS is 2%, the test CL system is currently operated at 10% fresh mix.

Gas samples were taken by bubbling gas mix in H₂O at four sampling spots of

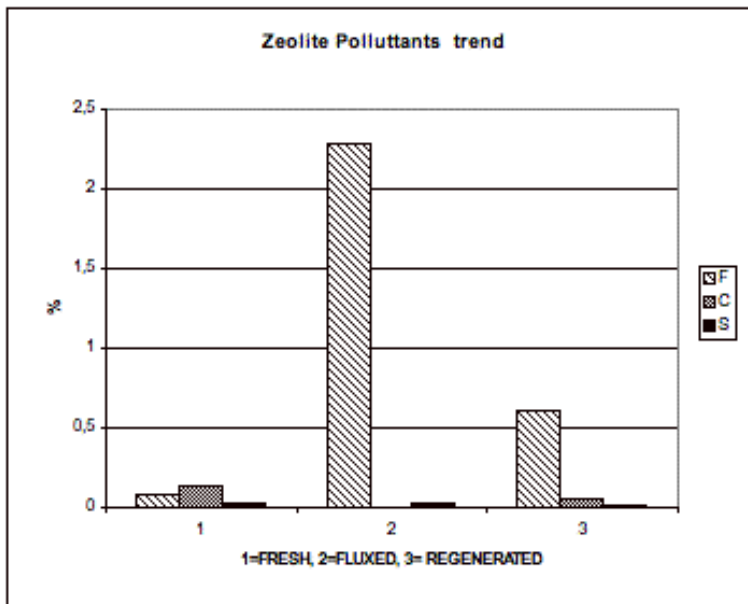


Figure 4: Zeolite trace elements trend (F, S, C).

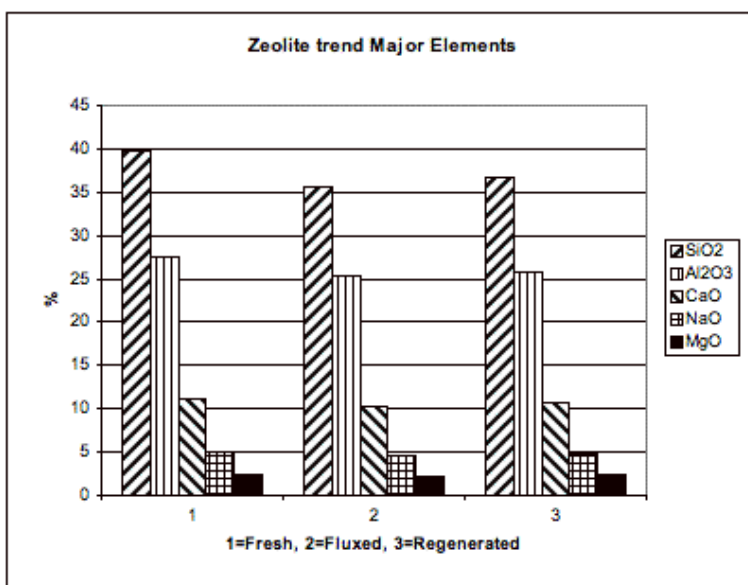


Figure 5: Zeolite major elements SiO₂, Al₂O₃, CaO, NaO, MgO.

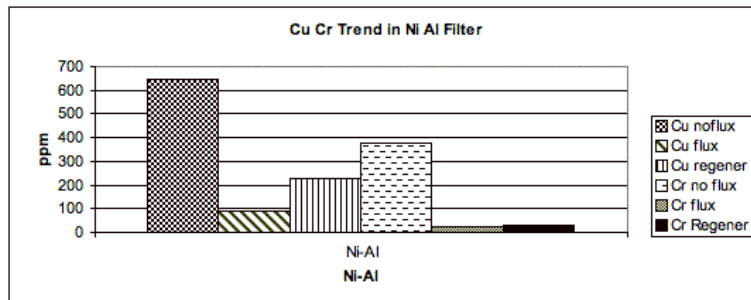


Figure 6: Ni-Al filter (Cu, Cr).

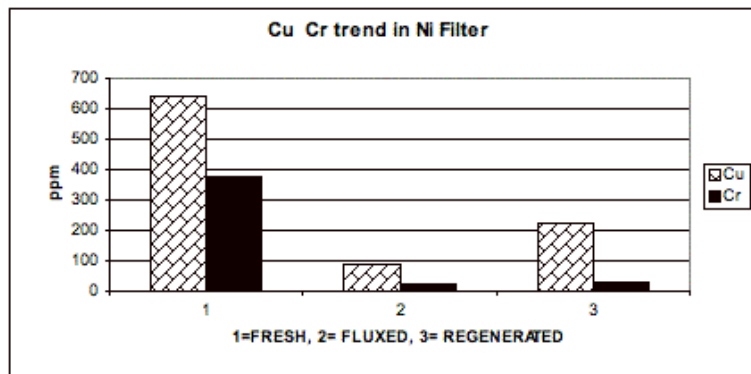


Figure 7: Ni filter (Cr, Cu).

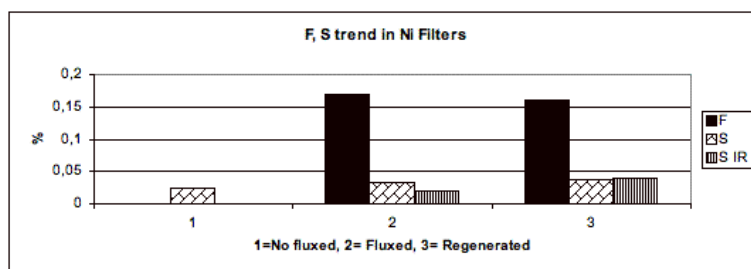


Figure 8: Ni filter (S-IR, S-TD/ICP, F).

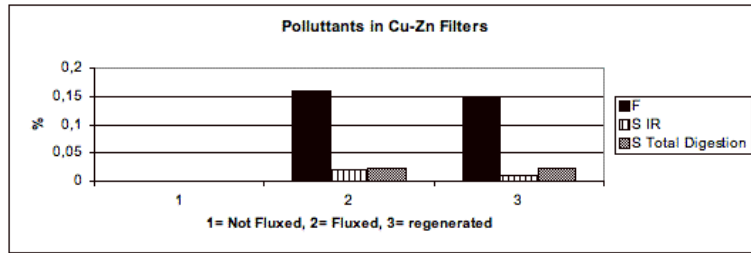


Figure 9: Cu-CuZn filters (S-IR, S-TD/ICP, F).

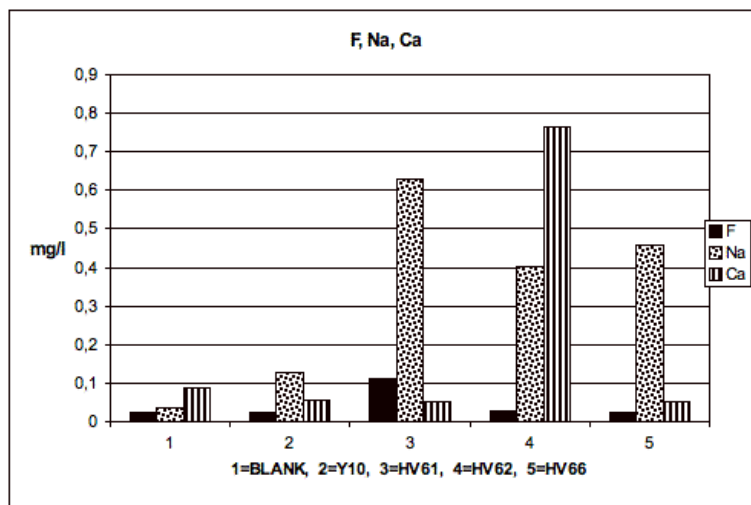


Figure 10: Gas concentrations F, Na, Ca.

CL, namely fresh mix, after gaps, after zeolite and after metallic filters for a total of approximately 20 days and a total volume of 1m^3 . Water solution was analyzed at HPLC.

Gas analysis in sampling points downstream of zeolite filters show Ca content increase in gas mixture, probably released by the 5\AA Ca zeolite type during filtration.

Results show presence of metals and F^- in gas mix (Fig. 10, 11, 12). We also monitored the pH of gas mixture finding confirmation for a buffering action of zeolite from pH 4.5 to pH 7 (Fig.13).

5 Bakelite

The material used for the containers of gas volume in CMS RPC detectors (called *bakelite*) is a plastic laminate obtained by wetting paper sheets with several kinds of resins. As

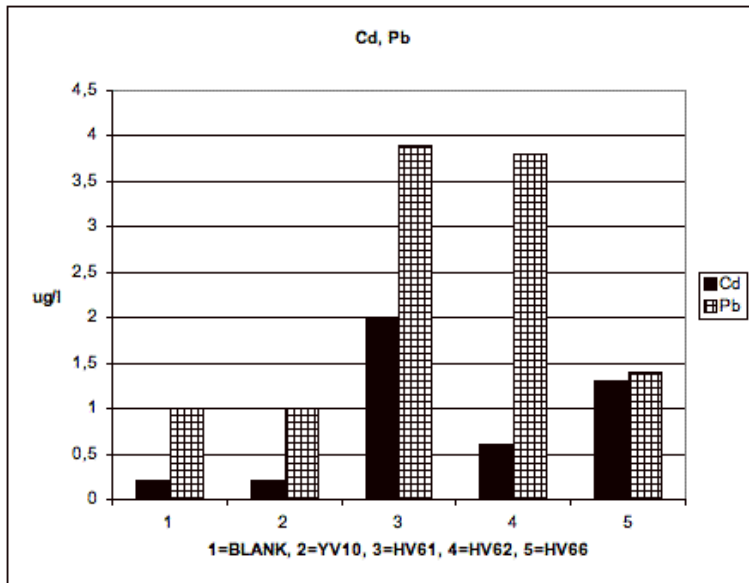


Figure 11: Gas concentrations Cd Pb.

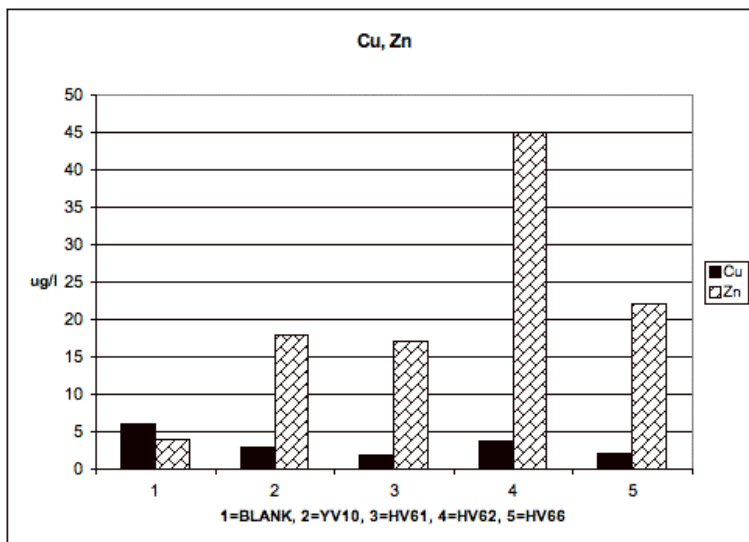


Figure 12: Gas concentrations Cu Zn.



Figure 14: Large gap exposed at GIF with defects and oil droppings.

such, bakelite is composed of many elements. Plastic laminates are produced by using mainly melaminic and fenolic resins. For the RPC gaps a laminate [24] composed of a bulk of paper wet by fenolic resin was used, surrounded by two outer layers of paper wet by melaminic resin, up to a 2mm thickness. In CMS RPC detectors, the inner layer is coated with linseed oil [26].

To study the effect of gas mixture on bakelite, samples of pure unoled bakelite (sample A) and of oiled bakelite (sample B) were taken from a gap irradiated at the GIF [8]. Gap was fluxed with the standard CMS RPC gas mixture in Closed Loop, and irradiated by a ^{137}Cs radioactive source at a total dose of 650 GBq.

To avoid the production of bakelite debris, the gap was open by blowing air until the spacer detachment. After separating the anode and cathode plates, defects have been marked on the bakelite inner surface. The defect areas of interest have been protected by adhesive guard rings to avoid the production of bakelite debris. A slow-motion drill with a 4cm-diameter tap was

used to sample discs for SEM analysis. Bakelite samples have been catalogued as Centre-Cell (CC) (undefected samples among spacers), Defect (D) where defects appear at visual inspection, and Manina (M) for areas close to spacers with linseed oil dripping.

SEM-EDS analysis was performed on unused, unoled bakelite samples (sample A), and on unused, oiled bakelite (sample B). Both samples A and B are unused, i.e., never fluxed with gas mixture, and are considered reference samples. Average composition

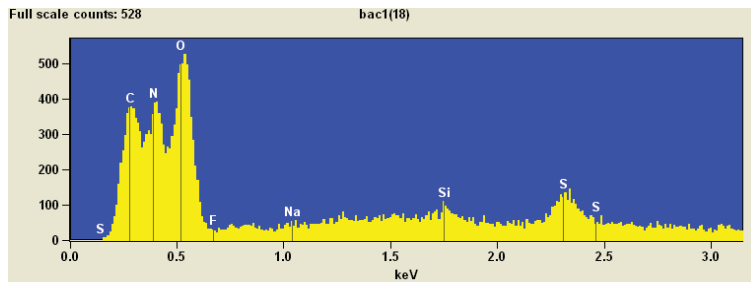


Figure 15: SEM-EDS of unused, unoiled bakelite (A).

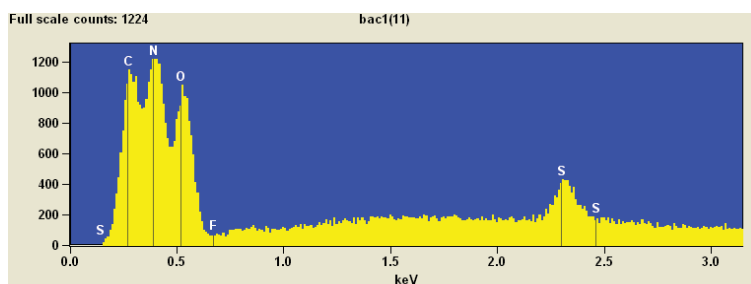


Figure 16: SEM-EDS of unused, oiled bakelite (B).

of samples A and B is shown in Fig. 15,16. SEM-EDS distributions are very similar, with the presence of linseed oil not being relevant, as they are formed being by the same components as bakelite. The N peak is consistent with the presence of melaminic resin in the outer bakelite layer. The composition of two fluxed bakelite samples from 50cm x 50cm gaps exposed in open loop (sample CC and sample D) is shown in Fig. 17,18.

The F peak is prominent, as well as the higher intensity of the Na peak, and the disappearance of the N peak. The defect areas are an erosion of the melaminic outer layer which allows one to access the composition of the inner fenolic layers, where N is absent. The presence of Na is explained with NaOH used as catalyzer in polymerization of resin, in amounts less than 1% for resin and less than 0.02% on the final product [24][25], as also suggested by similar results [7].

Observation under stereomicroscope of some defect areas showed their 3D morphology (Fig. 20). Online analyses at SEM-EDS allowed a study of composition of defect areas on a bakelite cross-section. Results showed the presence of Na and F . Mapping on these areas have shown concentration Fig. 6.1.6. Samples of defected areas were analyzed at XRD Fig. 21, showing a good match with the crystal structure of NaF.

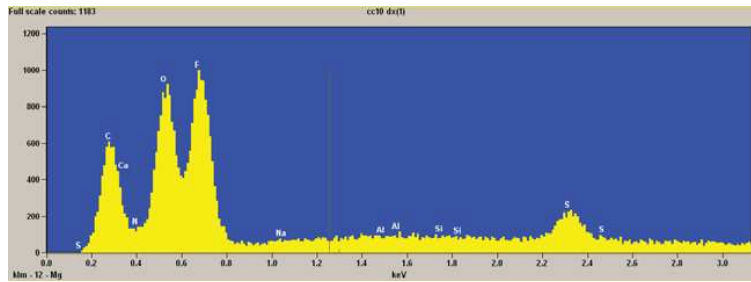


Figure 17: SEM-EDS of bakelite used, oiled centre-cell bakelite (CC).

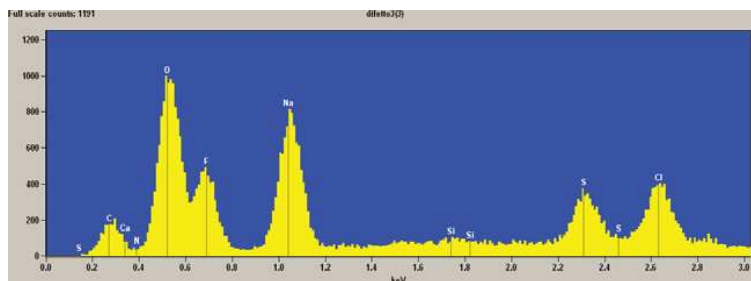


Figure 18: SEM-EDS of bakelite defect (D).

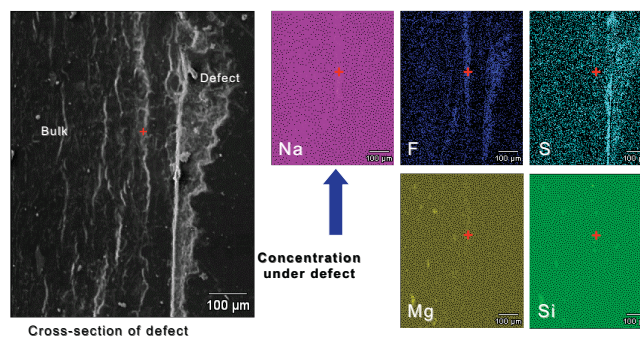


Figure 19: Cross-section of bakelite defect.

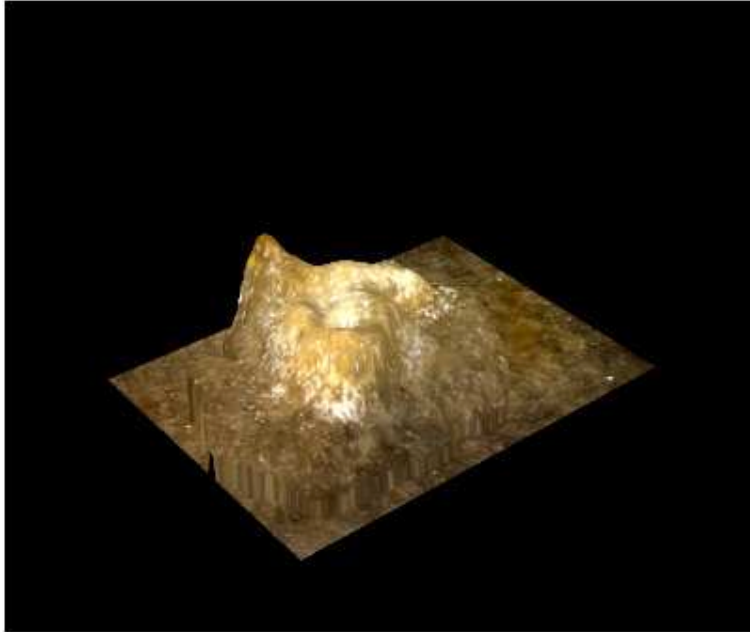


Figure 20: Macrophoto of defect.

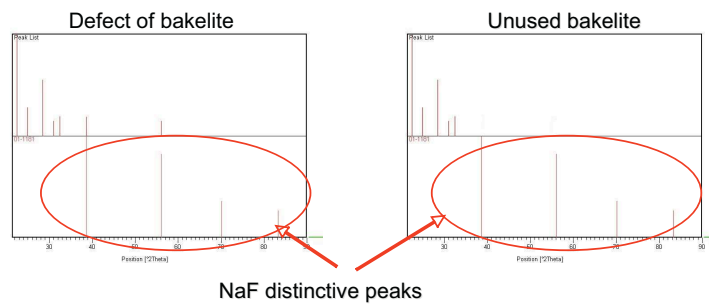


Figure 21: XRD showing NaF structure of defects.

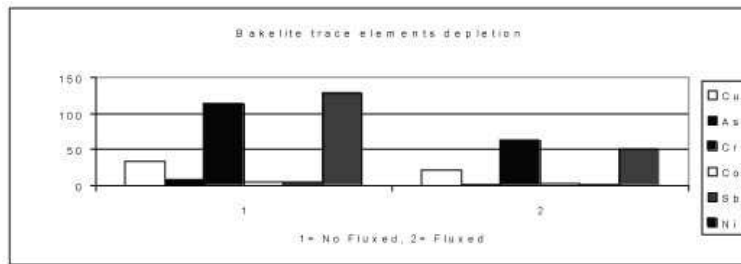


Figure 22: Bakelite concentrations (Cu, As, Cr, Co, Sb, Ni).

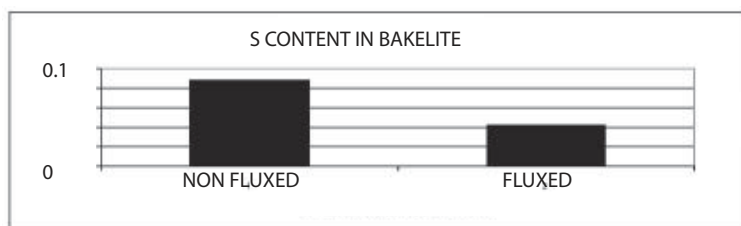


Figure 23: Bakelite concentration (S).

Chemical analyses have been performed to characterize bakelite composition of the trace elements. Trace elements may change depending on the production of bakelites. Fluxed and nonfluxed bakelites have been analyzed, results are shown in Fig. 22,23, several minor components are depleted in fluxed bakelites. A leaching effect is argued for Ni Cr Cu As Sb, that are therefore released in the system and in the RPC chambers. S is also depleted.

6 Conclusions and outlook

The purifiers currently used were selected originally in order to filter H₂O and O₂ while, given the gas mixture reactivity and the low pH, trace elements are released from purifiers and sent to the CL. The purifiers R3-12 and R3-11G are officially used to remove S and As from vapor or from solutions. Since the gas mixture used contains S, a part of it will interact with the filters depositing sulphides which will stay in the filters. Filters release trace elements naturally present in their unused composition (i.e., Cu and Cr in Ni-based filters). Ni-based filters trap S, very easily bound to Ni as a sulphide, which is not removed after regeneration.

Ni-Al₂O₃ filters release Cu and Cr during purification. They capture F and S, but

regeneration does not desorb filters. In Cu filters, S can easily form Cu and Zn sulphide, difficult to remove from filters. Both metallic filters capture F and S but the regeneration does not desorb them completely.

Zeolites captures F, Cu, Ni, S but regeneration does not fully desorb them. The gas mixture (due to the presence of HF) tends to leach SiO_2 and Al_2O_3 i.e. the framework of zeolite. Chemical analyses show a depletion in Si, Al contents in zeolites. A buffering effect is observed, with pH changing from 4.5 to 7.0 before and after the zeolite purifier.

The regeneration procedure used was not optimized for each filter. The regeneration procedure used is probably the one recommended by the zeolite producer. Purifiers Cu and CuO-ZnO are declared as nonregenerable [15], and the regeneration protocol followed (see §3) is not able to desorb the purifiers, as it is not sufficient to achieve complete Cu reduction which requires temperatures in the 350°C range. Elements such as As and S react with the Cu and Zn constituents in the catalyst to form stable metal complexes which do not migrate or desorb off of the catalyst. It is important to characterize the filters (both metallic and zeolite) in order to understand their selective absorption and ion-exchange possibility, their lifetime (in function of the complex system at CL), and finally it is of paramount importance to set up a specific desorption procedure.

The trace elements found clearly in the gas mixture are Cd, Pb, Zn. A lot of F, Na and Ca is also found. Zn could be released by zeolites, but no depletion is shown by zeolite analyses. Na is abundantly present in gas before filtering (probably released by the bakelite of RPCs), it decreases after the zeolite (being exchanged with Ca in the zeolite), but it stays abundant after the metallic purifiers. The presence of Ca in the gas mixture has implications that should be studied in more detail. Finally, the operation duration of R3-12 filters critically depends on the abundance of As and S. Full understanding of the CL chemistry calls for more detailed and systematic gas analyses.

In all fluxed bakelite samples we observe the presence of defects, in agreement with [7] [4]. Defects have been characterized as crystalline, identified as NaF. Bakelites release elements (Ni, Cr, Cu, Zn) due to a leaching process by the gas mixture. These elements can be filtered, and they actually are filtered by the zeolite which keeps such elements and undergoes successful regeneration for them. Zeolites are damaged by HF known to be produced in the gas mix, and the depletion of SiO_2 and Al_2O_3 measured by our analyses is a confirmation of such an effect. Measurements at XRD are planned to verify the zeolite structure after use. The bakelite samples analyzed show a depletion in some trace elements (Cu, Ni, As, Cr,Sb) after flux, with Cu and Ni possibly being adsorbed in zeolite.

Although this study explains some effects of the complex material science phenomenology in the RPC detector at the CL gas system, most of the basic questions on the

chemistry of gas and purifiers remain obscure. A systematic investigation is in progress at the ISR for chemical sampling of gas and purifiers, and correlation with currents behaviour in dedicated RPC detectors.

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