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Performance of the Prototype Gas Recirculation System with built-in RGA for INO RPC system

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Abstract

An open loop gas recovery and recirculation system has been developed for the INO RPC system. The gas mixture coming from RPC exhaust is first desiccated by passing through molecular sieve (3Å + 4Å). Subsequent scrubbing over basic active alumina removes toxic and acidic contaminants. The Isobutane and Freon are then separated by diffusion and liquefied by fractional condensation by cooling up to -26°C. A Residual Gas Analyser (RGA) is being used in the loop to study the performance of the recirculation system. The results of the RGA analysis will be discussed.

Key words: INO, R134a, RGA, SF\textsubscript{6}

1. Introduction

The Proposed INO RPC detector \cite{1} facility will contain nearly 27000 detectors of 2m X 2m size and will be continuously flushed with a gas mixture consisting of Isobutane, R134a(1,1,1,2 tetrafluoroethane), SF\textsubscript{6} (Sulphur hexafluoride) and Argon. The total internal volume of the detector stack will be in the range of 200 m\textsuperscript{3}. The prototype detector operates in avalanche mode with gas mixture R134a (95.0%), Isobutane (4.5%) and SF\textsubscript{6} (0.5%). The same prototype gas system can supply gas mixture suitable for streamer mode choosing Argon (30%), Isobutane (8%) and R134a (62%) \cite{2}.

To enhance the life and the performance of detectors, the gas content needs to be replaced at least once a day to keep harmful contaminants from accumulating. As direct consequence, 200 m\textsuperscript{3} of fresh gas mixture has to be fed into the detector stack and an equal volume has to be removed and safely disposed on daily basis. This operation involves very high operating cost and also causes the concentrations of SF\textsubscript{6} and R134a in air to exceed TLV (Threshold Limiting Value) to which a worker can safely be exposed for 12 hour a day) in the local working area. To overcome the problem of daily replenishment of fresh gas mixture, the gas mixture is reused by following methods:

2. Closed loop system

Gas mixture is re-circulated in RPC detectors and exhaust gas from RPC is purified to remove undesirable radicals and contaminants. Lost amounts of each gas component are measured continuously and topped up by gas flow systems. This technique involves maintaining precise pressure gradients across each element of the loop. Concentration changes at ppm level are to be measured. The system needs highly expensive measurement and control instrumentation.

3. Open loop system

In this method, component gases are separated from gas mixture by adsorption and condensation, purified and reused. During process of separation and condensation, the pressure, temperature and surface activation are maintained in such way that Isobutane is selectively adsorbed by the activated Palladium pellets in the first chamber and separated from the gas mixture by condensation while Freon...
R134a is carried out into the next chamber and condensed at the same pressure but at low temperature. Pressure versus condensation temperature curves are plotted in Fig. 1. Each curve represents vapour-liquid transition with respect to partial pressures and temperatures. For Isobutane, chamber temperature is typically -13°C at 0.5 bar. After Isobutane is removed, the remaining gas mixture flows into the next chamber where conditions (-26°C and 0.5 bar) are set for Freon R134a to condense. The residual gas mixture coming out of fractional condensation column is concentrated with Argon and SF₆ along with some trace vapours of Isobutane and Freon R134a.

4. The performance of Open loop system

The criteria for evaluating performance of open loop recovery system are:

4.1. Volumetric efficiency

Volumetric efficiency is defined as the ratio of gas volume collected in the system to gas volume entering into it, both measurements being at equal pressure and temperature. It indicates the residual volume of gas escaping the system. Presently bubbles are used to measure approximate volume flow rates of gas mixture entering the system, bypassing condensation chambers through safety vent and leaving the system as uncondensed gas mixture. It is observed that contamination due to radicals is nearly absent in the gas mixture coming out of the RPC detector. This could probably be due to low radiation intensity application. Major component of contamination is found to be water vapour which is completely removed by adsorption to a residual concentration of 10 ppm without any loss of gas mixture. Due to this reason the efficiency of volumetric recovery is found to be in the range of 85% to 95%.

4.2. Analysis of Gases: Measurement by RGA

A residual gas analyser (RGA) is a mass spectrometer of small physical dimension that can be connected to a vacuum system and whose function is to analyse the gas inside the vacuum chamber. A small fraction of gas molecules are injected into the chamber which are ionised (positive ions), and the resulting ions are separated, detected and measured according to their molecular masses. In the present case the purity of gases is measured by RGA, manufactured by Standford Research Systems (SRS) model RGA 300 probe[3]. The probe is interfaced with the chamber and turbo vacuum pump as shown in the Fig. 2. This analyser has a capacity to detect and measure up to mass number 300. Operating pressure for measurement is fixed at 3.5x10⁻⁵ mbar whereas background vacuum is at 1x10⁻⁷ mbar Fig. 3 shows the analysis of background gases.

Reference scans for each pure constituent gas is generated by sampling the gas directly from the cylinders. As the library does not contain for the R134a gas and Isobutane gas, scan for these gases were acquired and used as reference. It is seen that the plots for SF₆ gas does not fully agree very well with library values for pure gases. It is also seen that locally available R134a has adequate purity, as the RGA plot matches with high purity sample (Fig. 4).
5. Measurements and RGA analysis

5.1. Purity of Gases

- Sample reference of pure gases:
  - Fig. 5 is a normalised plot of mass versus relative abundance for pure gases considering main fragment as 100%. In pure spectra of R134a major fragments are in mass number (m/e) 33(CHF\textsubscript{2}F\textsuperscript{+}), 69(CF\textsubscript{3}+), 83(C\textsubscript{2}H\textsubscript{2}F\textsubscript{3}+) and 32(CHF\textsuperscript{+}). Similarly for Isobutane, the mass numbers are 43(C\textsubscript{3}H\textsubscript{7}+), 41(C\textsubscript{3}H\textsubscript{5}+), 42(C\textsubscript{3}H\textsubscript{6}+), 27(C\textsubscript{2}H\textsubscript{5}+) and for SF\textsubscript{6} are 127(SF\textsubscript{6}+), 89(SF\textsubscript{5}+) and 108(SF\textsubscript{4}+). The strong and weak characteristic peaks of each gas can be seen while other peaks arising from impurity of more than 1% concentration, are not apparent. These plots serve as reference.

- Comparison between samples taken at inlet and outlet of RPC detector
  - To estimate the extent of contamination entering of the gas mixture during its passage through detector due to discharge [4], is analysed at inlet and outlet of the RPC detector (AB13) over extended period of time. Variation of some major fragments for Isobutane (Fig. 7), SF\textsubscript{6} (Fig. 8) and R134a (Fig. 9) are shown, where in all these figures the horizontal scale represents the sample number. Fractional percentages are calculated with respect to the main peak (m/e=33) which corresponds to R134a(95% of the mixed gas). Effect due to ingress of external contamination is ruled out as some mass numbers show an increase of peak height at expense of others. Indications are that some fragments are created inside RPC during the passage of gas mixture. Fig. 10 is a plot of certain radicals and contaminants generated during those periods.

- Samples of recovered gas by open loop method
  - Samples of recovered gas after condensation show concentration of Isobutane rise from 4.5% in the input gas mixture to 95% as separated gas phase. However Freon is extracted from 95% in the gas mixture to 98% in the separated state (Figure. 11). There appears to some leftover Isobutane entering into Freon recovery as cross contamination. This is probably due to saturation of catalyst due to excessive flow.
6. Problems faced in open loop recovery method

- Air hinders condensation process by greatly reducing partial pressures of gases. It is therefore necessary to remove traces air completely from the loop either by evacuation or long cycle purging. Air concentration not more than 0.2% is acceptable for recovery process to be effective.
- Palladium catalyst is sensitive to presence of Hydrogen Sulphide. These contaminants render the catalyst by passivation of surface. Such contaminants may be produced during $SF_6$ breakdown and get carried up to the catalyst. Suitable chemical traps are provided at the input of the recovery system but their life is limited and should be replaced from time to time. The catalyst can also be regenerated at $1000^\circ C$ under oxidizing atmosphere. To regain surface activity catalyst is reduced by Hydrogen at $1000^\circ C$.
- Disturbances in pressure balance: Pressure unbalance occurs when total flow rate changes transient condition where excess gas flows into vent thus loss of efficiency of recovery. This problem can be solved by using active smart pressure and flow controllers.

6.1. Closed loop Gas Recirculation system

Since it is found that only small quantity contaminants is occasionally generated, a closed loop system is built with minimum instrumentation to study effect of accumulation of radicals and contaminants. Fig. 12 is layout of closed loop system.

During the passage of gas mixture through the detector, a test detector AB13 is used to evaluate gas mixture while it is maintained under operating conditions with a fixed quantity of gas re-circulating in the loop. The accumula-
...tion of contamination is monitored from time to time by sampling into RGA to check amount of atmospheric impurities entering the gas mixture through detector body or other connections in the loop. Gas mixture is once filled in to a ballast chamber having 5 litre capacity. A diaphragm pump displaces gas through the loop without creating a negative pressure inside RPC. The quality of the gas mixture is monitored with respect to change in parameters of the detector. Fig. 13 shows variation in leakage current and lab temperature with time. The contribution from ambient temperature change during this period is found to be quite small (Fig. 13). The increase in leakage current and noise rate is related with amount of air and moisture entering during pumping cycles. It can also be seen that the effect is cumulative.

7. Conclusion

(i) The open loop system is able to extract Isobutane with good purity and minimal cross contamination from Freon or other gaseous component.

(ii) Freon recovery is sensitive to capacity of adsorption by catalyst. Excess Isobutane is carried into Freon recovery and reduces purity of Freon.

(iii) The formation of Radicals during RPC operation under present condition is a rare event.

The leak rate under operating conditions governs stability of RPC performance under close loop recirculation process.

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