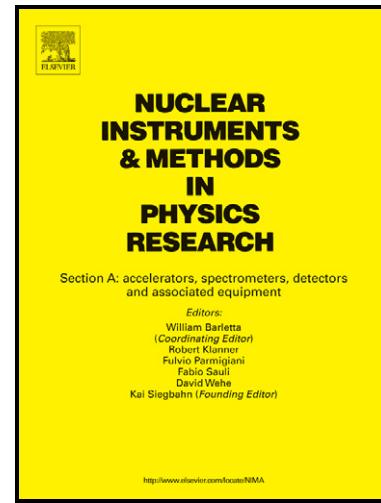


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Performance of the Prototype Gas Recirculation System with built-in RGA for INO RPC system

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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, SF₆

1. Introduction

The Proposed INO RPC detector [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 tetrafluoro ethane), SF₆ (Sulphur hexafluoride) and Argon. The total internal volume of the detector stack will be in the range of 200 m³. The prototype detector operates in avalanche mode with gas mixture R134a (95.0%), Isobutane (4.5%) and SF₆ (0.5%). The same prototype gas system can supply gas mixture suitable for streamer mode choosing Argon (30%), Isobutane (8%) and R134a (62%) [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 m³ 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 SF₆ and R134a in air to exceed TLV (Threshold Limiting Value: to which a worker can safely be exposed for 12 hours

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

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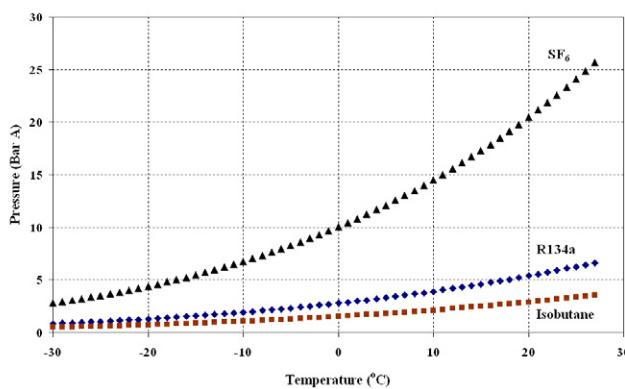


Fig. 1. Vapor Pressure versus condensation temperature

43 R134a is carried out into the next chamber and condensed
 44 at the same pressure but at low temperature. Pressure ver-
 45 sus condensation temperature curves are plotted in Fig.
 46 1. Each curve represents vapour-liquid transition with re-
 47 spect to partial pressures and temperatures. For Isobutane,
 48 chamber temperature is typically -13°C at 0.5 bar. After
 49 Isobutane is removed, the remaining gas mixture flows into
 50 to next chamber where conditions (-26°C and 0.5 bar) are
 51 set for Freon R134a to condense. The residual gas mixture
 52 coming out of fractional condensation column is concen-
 53 trated with Argon and SF₆ along with some trace vapours
 54 of Isobutane and Freon R134a.

55 4. The performance of Open loop system

56 The criteria for evaluating performance of open loop re-
 57 covery system are:

58 4.1. Volumetric efficiency

59 Volumetric efficiency is defined as the ratio of gas volume
 60 collected in the system to gas volume entering into it, both
 61 measurements being at equal pressure and temperature. It
 62 indicates the residual volume of gas escaping the system.
 63 Presently bubbles are used to measure approximate vol-
 64 ume flow rates of gas mixture entering the system, bypass-
 65 ing condensation chambers through safety vent and leav-
 66 ing the system as uncondensed gas mixture. It is observed
 67 that contamination due to radicals is nearly absent in the
 68 gas mixture coming out of the RPC detector. This could
 69 probably be due to low radiation intensity application. Ma-
 70 jor component of contamination is found to water vapour
 71 which is completely removed by adsorption to a residual
 72 concentration of 10 ppm without any loss of gas mixture.
 73 Due to this reason the efficiency of volumetric recovery is
 74 found to be in the range of 85% to 95%.

75 4.2. Analysis of Gases : Measurement by RGA

76 A residual gas analyser (RGA) is a mass spectrometer of 97
 77 small physical dimension that can be connected to a vac-

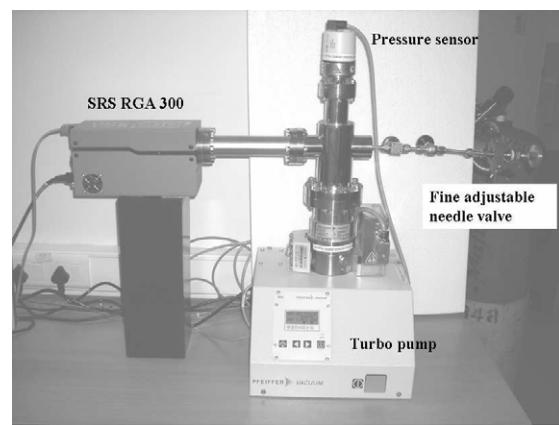


Fig. 2. RGA Setup

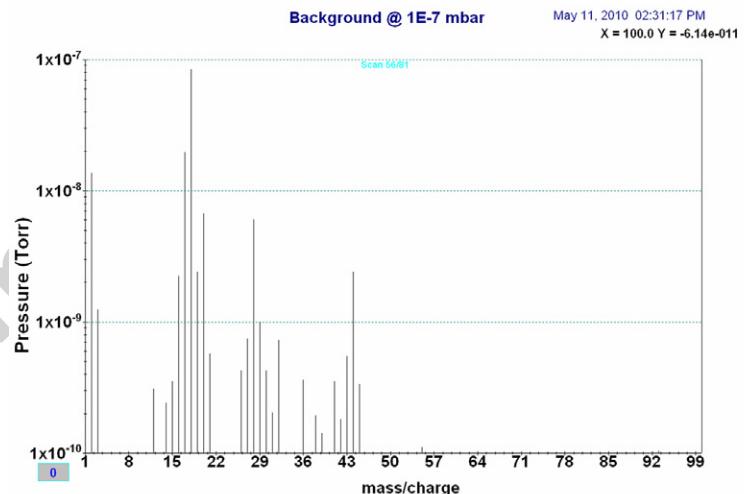


Fig. 3. Background mass spectrum

uum 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 3.5×10^{-5} mbar where as background vacuum is at 1×10^{-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).

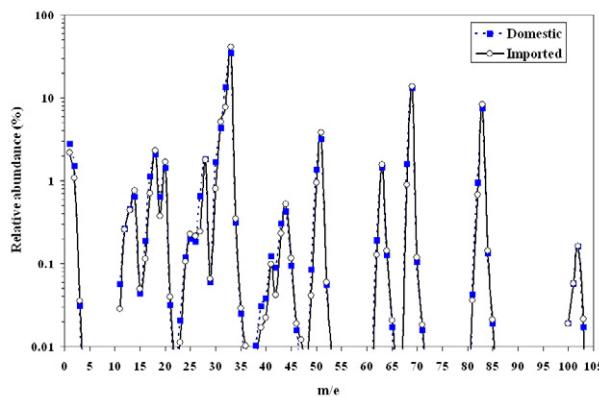
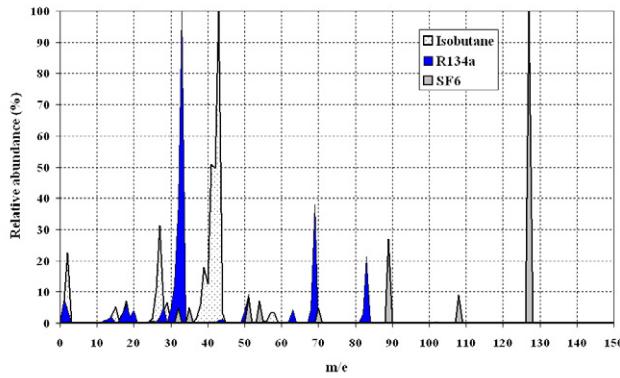


Fig. 4. Comparision of different samples of R134a

Fig. 5. RGA spectra of pure gases. Main fragment of Isobutane, R134a and SF₆ are 43, 33 and 127 respectively.

99 5. Measurements and RGA analysis

100 5.1. Purity of Gases

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- 101 – Sample reference of pure gases: 125
 - 102 · Fig. 5 is a normalised plot of mass versus relative 126 abundance for pure gases considering main fragment¹²⁷ as 100%. In pure spectra of R134a major fragments¹²⁸ are in mass number (m/e) 33(CH₂F⁺), 69(CF₃⁺),¹²⁹ 83(C₂H₂F⁺) and 32(CHF⁺). Similarly for Isobutane¹³⁰ the mass number are 43(C₃H₇⁺), 41(C₃H₅⁺), 42(C₃H₆⁺)¹³¹ and 27(C₂H₃⁺) and for SF₆ are 127(SF₅⁺), 89(SF₃⁺)¹³² and 108(SF₄⁺). The strong and weak characteristic¹³³ peaks of each gas can be seen while other peak arising¹³⁴ from impurity of more than 1% concentration, are not¹³⁵ apparent. These plots serve as reference. Fig. 6 is a¹³⁶ spectra for Isobutane taken at different pressures in¹³⁷ cluding the library spectra of butane for comparison.¹³⁸ At higher vacuum, the effect of background gases such¹³⁹ as Oxygen and moisture entering from the pumping¹⁴⁰ system is high. But the total contribution of impurity¹⁴¹ is less than 1%.
 - 113 – Comparison between samples taken at inlet and outlet¹⁴³ of RPC detector 144
 - 114 · To estimate the extent of contamination entering of the¹⁴⁵

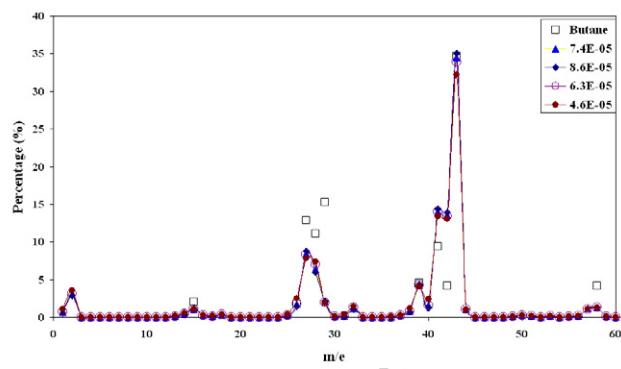


Fig. 6. RGA spectra of Isobutane in different pressure. In the legend quoted pressures are the operating pressures in mbar.

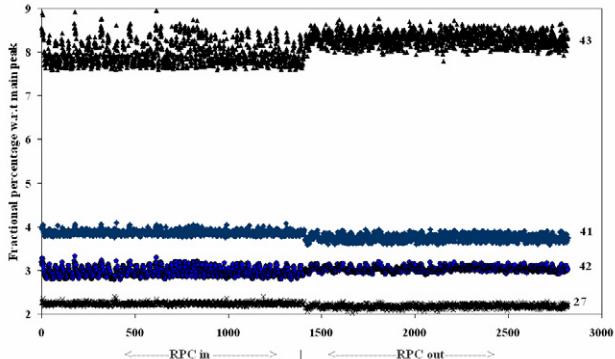


Fig. 7. Major fragments of Isobutane. The numbers (27, 41, 42 and 43) indicate mass number of major fragments.

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), SF₆ (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.

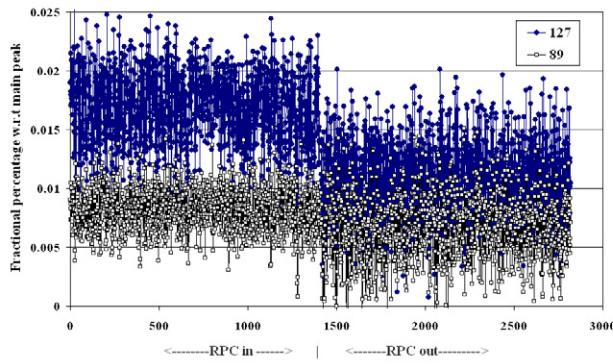
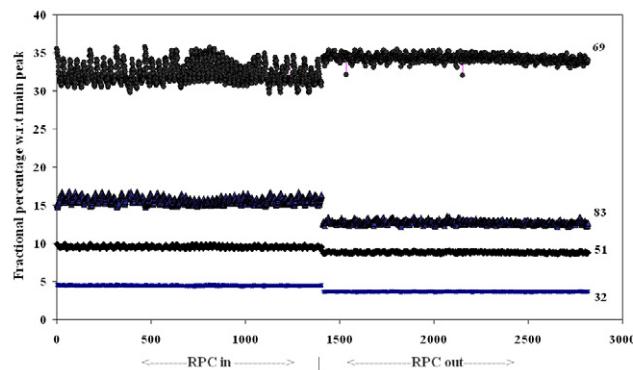
Fig. 8. Major fragments of SF_6 

Fig. 9. Major fragments of R134a. The numbers (32, 51, 69 and 83) indicate mass number of major fragments.

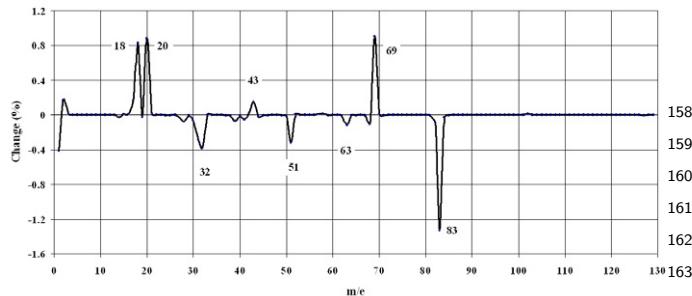


Fig. 10. Percentage change of various radicals (Output-Input)

146 6. Problems faced in open loop recovery method

- 147 – Air hinders condensation process by greatly reducing¹⁶⁸
148 partial pressures of gases. It is therefore necessary to re-
149 move traces air completely from the loop either by eva-¹⁶⁹
150 cuation or long cycle purging. Air concentration not more¹⁷⁰
151 than 0.2% is acceptable for recovery process to be effec-¹⁷¹
152 tive.¹⁷²
- 153 – Palladium catalyst is sensitive to presence of Hydrogen¹⁷³
154 Sulphide. These contaminants render the catalyst by pas-¹⁷⁴
155 sivation of surface. Such contaminants may be produced¹⁷⁵
156 during SF_6 breakdown and get carried up to the cata-¹⁷⁶
157 lyst. Suitable chemical traps are provided at the input of¹⁷⁷

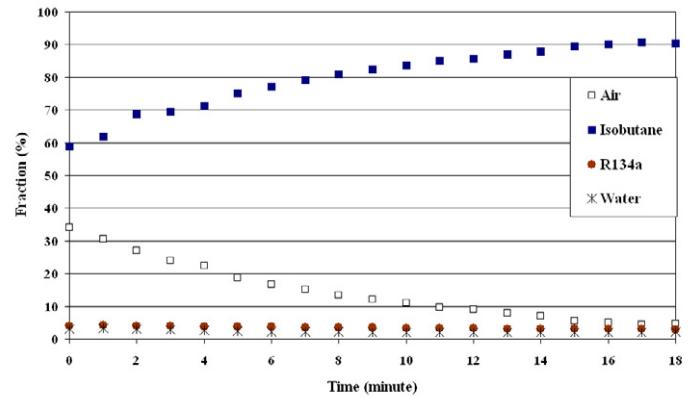


Fig. 11. Separation of Isobutane from mixture

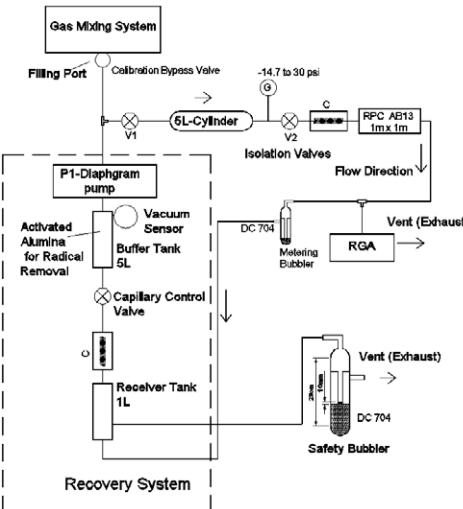


Fig. 12. Schematic of close loop system

the recovery system but their life is limited and should be replaced from time to time. The catalyst can also be regenerated at 1000°C under oxidizing atmosphere. To regain surface activity catalyst is reduced by Hydrogen at 1000°C [5].

– 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.

146 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-

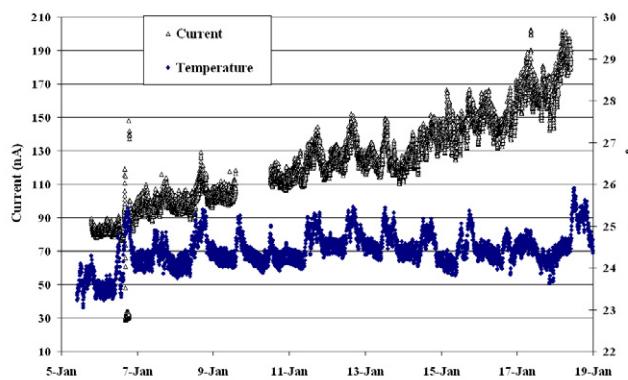


Fig. 13. Variation of current of AB13 and lab temperature

178 tion of contamination is monitored from time to time by
 179 sampling into RGA to check amount of atmospheric impu-
 180 rities entering the gas mixture through detector body or
 181 other connections in the loop. Gas mixture is once filled in
 182 to a ballast chamber having 5 litre capacity. A diaphragm
 183 pump displaces gas through the loop without creating a
 184 negative pressure inside RPC. The quality of the gas mix-
 185 ture is monitored with respect to change in parameters of
 186 the detector. Fig. 13 shows variation in leakage current and
 187 lab temperature with time. The contribution from ambient
 188 temperature change during this period is found to be quite
 189 small (Fig. 13). The increase in leakage current and noise
 190 rate is related with amount of air and moisture entering
 191 during pumping cycles. It can also be seen that the effect
 192 is cumulative.

193 7. Conclusion

- 194 (i) The open loop system is able to extract Isobutane
 195 with good purity and minimal cross contamination
 196 from Freon or other gaseous component.
- 197 (ii) Freon recovery is sensitive to capacity of adsorption
 198 by catalyst. Excess Isobutane is carried into Freon
 199 recovery and reduces purity of Freon.
- 200 (iii) The formation of Radicals during RPC operation un-
 201 der present condition is a rare event.

202 The leak rate under operating conditions governs stabil-
 203 ity of RPC performance under close loop recirculation pro-
 204 cess.

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