Deuterium Gas Analysis by Residual Gas Analyzer

B K Das*,1, R Shukla1, R Das1, A Shyam1 and A D P Rao2

1Energetics & Electromagnetics Division, Bhabha Atomic Research Centre, Autonagar, Visakhapatnam, India
2Nuclear Physics Department, Andhra University, Visakhapatnam, India

Corresponding author’s e-mail: dasbabu31@gmail.com

Abstract. Hydrogen gas is generated by electrolysis method in a compact hydrogen generator. A simple procedure reduces handling and storage of hydrogen cylinders for laboratory applications. In such a system, we are producing deuterium gas from heavy water by electrolysis method. After production of the deuterium gas, we have checked the purity level of the outgoing deuterium from the electrolyser. The test was carried out in a high vacuum system in which one residual gas analyser (RGA) was mounted. The deuterium gas was inserted by one manual gas leak valve into the vacuum system. In this study, the effect of the emission current of the RGA on the detection of the deuterium was performed. In this paper, we will discuss the detail analysis of the deuterium gas and the effect of the emission current on the partial pressure measurement.

1. Introduction
Use of modern available technology for the basic requirements in research and development laboratories becomes important due to various safety reasons. One of the most important safety issues is handling of pressurized gas cylinders specifically for the flammable gases. Handling and use of hydrogen cylinders in laboratories requires lot of precautions. To avoid such safety issues related to handling, storage and use of hydrogen cylinders, we are working on one electrolysis system [1] for generation of hydrogen isotopes. Deuterium gas is generated by this electrolysis system from heavy water. The purity of the gases produced by this system is important for our application. The purity of the deuterium gas produced by an electrolysis system in our laboratory was checked in a high vacuum system by residual gas analyzer. In the next sections we will discuss about the detail experimental set up and the results.

2. Experimental set up
We are using one compact electrolysis system purchased from M/s. PCI Analytical Pvt. Ltd [2]. The photograph of the system is shown in figure 1. The electrolysis flow diagram is shown in figure 2. Potassium Deuteroxide (KOD) is used as electrolyte and heavy water. The usage of voltage across the electrolyte results in hydrolysis, breaking down the heavy water molecule into deuterium and oxygen gas, which are separated by the gas permeable membrane. To achieve the desired level of purity, the deuterium gas goes through series of purification and moisture removal systems while the oxygen gas is being discharged into the atmosphere. The electrolytic membrane technology has its own benefits over alternative hydrogen gas generator techniques as it is clean and requires minimum maintenance.
The deuterium gas thus produced by the electrolysis system is tested by one residual gas analyzer (RGA), Hiden Analytical Ltd., UK [3] make, mounted on a high vacuum system pumped by turbo molecular pump as per the figure 3. A vacuum in the order of 3.5E-7 Torr was achieved without baking. The deuterium gas was inserted into the chamber by one manual gas leak valve. The basic principle of the RGA is based on ionize and measurement [4]. RGA ionizes the neutral gases and then measure the ion current. The ion current directly corresponds to the number of neutral molecules of that particular ion. Neutral molecules present inside the vacuum chamber are ionized by one electron impact ion source present inside the RGA assembly. Once the gas is ionized they are focused to one filter system made of four electrodes know as quadruple. The quadruple filters the ions on the basis of their mass to charge ratio. The quadruple is powered by RF and DC voltages. The RF power makes the ions to oscillate inside the quadruple where as the DC voltage absorbs the unwanted ions when they strike any of the electrode surfaces. The ions those are passed through the quadruple are detected at the end either by one faraday cup or by channel electron multiplier. The RGA was operated in profile mode and MID mode. In profile mode, RGA does series of scans for each mass number. The scans were performed for different emission current of the filament. The emission current decides the ionization probability of the neutral gas molecules in the vicinity of the ion source.

Fig. 1. Photograph of Hydrogen Generator

Fig. 2. Flow Diagram of Hydrogen Generator
3. Results

With the above experimental set up different spectrum were obtained from RGA for deuterium concentration, it’s stability and the effect of emission current on the deuterium measurement. The spectrum obtained with the background i.e. without the filling of deuterium is shown in figure 4. From this figure, it could be observed that the vacuum system is free from leaks. The Oxygen signal at mass number 32 is absent, the nitrogen signal at mass number 28 is very less and there is no peak at mass number 14. The peak at mass number 28 is a contribution from carbon monoxide, carbon dioxide and nitrogen. As the vacuum system is not exposed to any heat, presence of water vapor in the system is unavoidable. The peak at mass number 18, 17, 2 and 1 are due to the presence of water vapor in the system.

![Photograph of Experimental Set Up](image)

**Fig. 3.** Photograph of Experimental Set Up

![Spectrum of profile scan of RGA for background](image)

**Fig. 4.** Spectrum of profile scan of RGA for background
The spectrum shown in figure 5 is obtained with the deuterium filled pressure at 2.5E-5 Torr. The peak at mass number 4 is due to deuterium. In the spectrum there is no other significant peak is shown. There is a very little peak at mass number 3, which is due to formation of DH (Deuterium Hydride). This may be due to the presence of residual hydrogen left behind while the system was tested for hydrogen production. The formation of DH molecule may disappear after long use of the system for the production of deuterium. Another very little peak at mass number 18 is due to the presence of water vapor in the vacuum chamber even before filling of the deuterium gas. The spectrum shows free from any shorts of impurities.

The electrolysis system continuously generates deuterium. To check the stability of the production of the deuterium, one MID scan was performed with the RGA. In MID scan, RGA continuously scans the particular mass number for given period of time. Such a scan is performed simultaneously for deuterium, oxygen, nitrogen and water vapor. Deuterium is for it’s stability check and other molecules for their appearance during deuterium production either from electrolysis or from any other sources. Such a spectrum is shown in figure 6. The red line corresponds to deuterium where as the other colored lines for rest of the molecules. The red line once it is stable after production is steady throughout the measurement period for duration of ten minutes.

In an effort to check the effect of electron emission current on the partial pressure measurement, one measurement was done for different emission current. The electron emission current helps for the efficient ionization of the neutral gases in RGA. The spectrum shown in figure 7 represents the effect of emission current on the partial pressure measurement of deuterium. Though here, deuterium gas was taken as the test gas, the result may be applicable to any other gases. There is an exponential growth of the partial pressure with the emission current. The measurement was done at filled pressure of 4.5E-5 Torr. It is observed that the more emission current, i.e. higher side of the instrument specification is giving correct measurement, where as the lower side of the emission current is giving more incorrect value. The idea of using the instrument with lower emission current may be helpful for suppression of background.
Fig. 6. MID scan of RGA for stability test of the deuterium production

Fig. 7. Deuterium partial pressure Vs electron emission current
Conclusion
In this work, the production of deuterium gas by compact electrolysis system was carried out. The purity of deuterium is checked by residual gas analyzer. The output deuterium from the electrolysis system was found to be ultra pure and stable. Residual impurities are absent. This type of system for deuterium/hydrogen production in laboratories is useful from safety and logistic point of view. The emission current of RGA should be at the specified value according to the supplier’s specification. Any lower value may give incorrect measurement.

References
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