iJRASET
International Journal For Research in
Applied Science and Engineering Technology

INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 6 **Issue:** IV **Month of publication:** April 2018

DOI: http://doi.org/10.22214/ijraset.2018.4692

International Journal for Research in Applied Science & Engineering Technology (IJRASET)
ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 6.887
Volume 6 Issue IV, April 2018- Available at www.ijraset.com

Studies on Design, Fabrication and Assembly of Quadrupole Mass Spectrometer

Hariram K Unnithan[1], Arun Kumar G[2]
[1]*PG Scholar, Department of Mechanical Engineering, Sri Vellappally Natesan College of Engineering*
[2]*Asst.Professor, Department of Mechanical Engineering, Sri Vellappally Natesan College of Engineering*

Abstract: Quadrupole mass spectrometer is an extremely versatile analytical instrument with applications in wide areas of science and technology like gas analysis, residual gas analysis and compositional analysis of fuel gases etc. QMS consist of electron bombardment type ion optics system, quadrupole based analyser system and a collector system consists of multiplier. Design of some subsystems have been modified and has higher ultimate vacuum in system, mass range, resolution (resolving power) in addition to compactness, superior aesthetics easier operation and maintenance. High care is needed for fabrication and assembling of components because the performance depends on the accuracy of the subsystems. This paper explains the details regarding the design, fabrication and assembly of various subsystems of QMS that improves the performance in terms of sensitivity and precision.
Keywords: QMS, Q-Rod, Multiplier, Vacuum chamber, Radio frequency

I. INTRODUCTION

Due to its high sensitivity, selectivity, versatility combined with excellent precision and accuracy, mass spectrometry has become an essential analytical technique for variety of applications in the nuclear fields [1], [2]. Mass Spectrometry is mainly based on the motion of a charged particle in a magnetic field or electric field [3], [4], [5]. Mass spectrometers consist of a vacuum system which is used to maintain very high vacuum (negative pressure) required for the operation. This high vacuum is mainly for minimizing ion-molecule interaction, scattering and neutralization of the ions. A quadrupole mass analyser [25] consists of four electrically isolated hyperbolic or circular rods linked to RF and DC voltages. The combination of RF and DC voltages creates a region of strong focusing and selectively known as hyperbolic field. The main working principle of quadrupole mass spectrometer [9], [24] is ionization of neutral atoms or molecules of analyte by collision with the energetic electrons. The quadrupole mass spectrometer uses electron impact /bombardment ion source [10], [24]. The ion source of QMS consists of an ionization box which incorporates tungsten filament for producing electrons. The gas particles are introduced into the ion source; the electrons undergo inelastic collision with gas molecules and ionize them. The type of analyser used by QMS is radio frequency quadrupole. The type of the detector used in QMS is electron multiplier detectors [25]. It is capable of measuring very small ions currents that is below 10-14 A.

II. LITERTURE REVIEW

Literature survey includes the studies of design modifications, ion optics, collector system, analyser, vacuum chamber.

1) *Ion Source*: H.L. Brown, et al. studied various ion generation techniques. Studies deals with production of super excited neutrals on the counter electrode which then auto ionize in the R.F field and are subsequently detected by the multiplier. This source is on the analyser axis and close to the rod structure [19].
2) *Analyser*: W.M. Brubaker, et al. conducted experiment between round rods and hyperbolic rods and found that there was an improvement of a factor of two in resolution when hyperbolic replaced circular surfaces. The value in practice of the hyperbolic surfaces is questionable because the greater difficulties in machining and mounting these electrodes are likely to give both large and asymmetrical misalignment errors [23].
3) *Collector:* J.M. Goodings, et al. studied the addition of multiplier and obtained that the performance can be improved by using an electron multiplier as the collector in place of the faraday cup. In this multiplier is placed at right angles to the central axis or set back in a side tube [17].

III. DESIGN MODIFICATIONS

The design of QMS chamber, ion source including filament assembly and ion optics were modified to facilitate easy and faster assembly and better performance. The filament assembly employed in QMS is for the production of electrons. A modified filament assembly with ceramic (fired alumina) was developed. It is evaluated and was found to be better than conventional filament assembly in terms of stability and durability. For Ion optics assembly, the design is modified with a part of mesh type, so that the gas molecules can come out through any direction. The electrons produced from the filament will be bombarded on gas molecules and more amounts of ions will produce for the given samples. For vacuum chamber, the older S.S chamber is replaced by monolithic aluminium square chamber which is open at the top portion. In this top open design, the top portion is then covered by tempered glass and O-ring groove is provided for fixation of gasket for proper sealing of the vacuum chamber. The main advantage of this design is that the assembly of the components inside the chamber become very easy and no complications like rubbing or hitting within the side walls will take place. The components can be assembled individually to the chamber. Aluminium is a better material for vacuum because of its less out-gassing.

IV. DESIGN CONSIDERATIONS OF MAJOR COMPONENTS

A. Ion Source

The overall design features of main sub systems in QMS include ion source, analyser, collector system and vacuum system. The ionization is based on the principle of ionization of neutral atoms or molecules of analyte by collision with the energetic electrons. A proportion of the ions formed inside the cylindrical grid cage are extracted and focused into an approximately parallel beam by negative electrode plates. As it is desirable to direct the maximum ion flux into the quadrupole field, the ion apparatus is usually made as large as possible, consistent with the geometry of the rod assembly. The design of source is such that it can easily be dismantled and reassembled for cleaning. Other components coming under ion source side are the pre-filter for producing oscillatory field and to avoid the fringe field, source mount for mounting the ion source assembly and a source insulator to provide insulation between the source and the mount.

B. Analyser

The analyser consists of a parallel array of four rod electrodes mounted in square configuration. The ideal geometry dictates that each electrode should be hyperbolic in cross section but in practice, for ease of manufacture, round cylindrical rods often are employed. The field within the analyser is created by coupling opposite pairs of rods together and applying RF and DC potentials between pairs.Ions with selected m/q ratio will only follow stable trajectories and other follows unstable trajectories and tend toward infinite displacement. Ions that are successfully transmitted through the analyser are said to possess stable trajectories and these are recorded by detector system. The entire quadrupole mass analyser system is housed in aluminium chamber with entrance and exit apertures for ions. Major part of analyser system is the quadrupole rod. The length and diameter of the rods are designed on the basis of the type of application to which they are used for and the operating RF voltage, Mass number range and resolution. It is designed with high accuracy for better performance of the QMS. Another major part is the Q-Rod mount, which is designed for the purpose of holding the quadrupole rods. 4 semi-circular cuts are designed on the Q-rod mount as shown in the figure for the passage of RF field around the rods, as the RF field generated will move in a circular path with respect to the rods and the radius of the semi-circular cuts are designed in accordance with the diameter of the rod. Next is the ceramic mount, mainly an insulating material which is used to separate two conducting material (Q-Rod & Q-Rod mount). Here the four Q-rods are assembled into the ceramic .Last part is the shield, designed mainly to attract the positively charged ions that are coming through the analyser and then pass into the multiplier for collection.This is designed with a structure similar to that of the Q-rod mount and is of very small thickness.

C. Collector

Ion detector represent the simplest, cheapest and most reliable means for detecting the ion beams emerging from the exist aperture of the quadrupole. Earlier faraday cup or plate used in conjunction with a sensitive electronic amplifier was used as the ion detector in QMS. But this has a relatively high noise level and slow response. These drawbacks are avoided by using an electron multiplier as the detector in place of the faraday cup. Both the signal to noise ratio and the speed of response of electron multiplier is much better than the faraday cup and it is able to detect very low ion currents. The major advantages of the detectors are their high gain, high dynamic range and fast response. The multiplier is mounted to the chamber using a multiplier mount.

D. Outer chamber

QMS works in vacuum to provide free path for the transmission of ions as per the requirement of the experiment. This requires the use of specially made vacuum chambers and vacuum pumping systems. The material selected for QMS outer chamber/vacuum chamber is Aluminium. This is because out gassing rates are excellent and even better than stainless steel. Weight of envelope is less due to low density. However, more thickness is required for fabrication due to its poor strength. A vacuum vessel requires many joints and vacuum tight sealing is required in these joints. Different sealing materials such as elastomers and metal gaskets are used for this.

E. Supports

The whole QMS vacuum chamber with the assembly of all the components is needed to fix on table top. Supports are designed for supporting the chamber above the table top so that the visibility of the mass spectrometer can be improved.

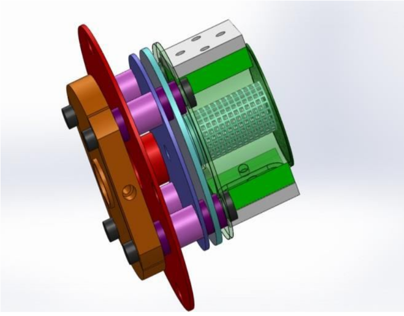


Fig. 1 Ion Source

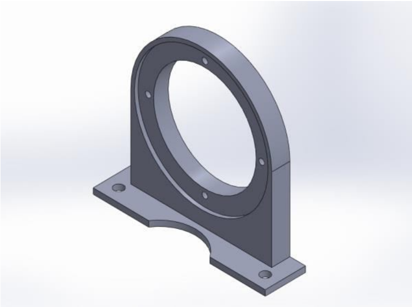


Fig. 2 Source Mount

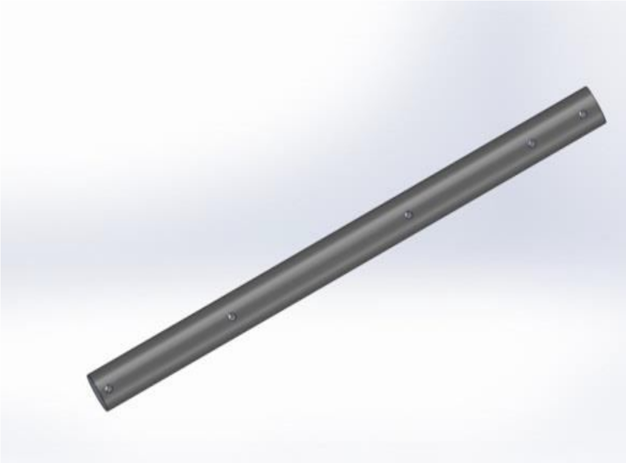


Fig. 3 Q-Rod

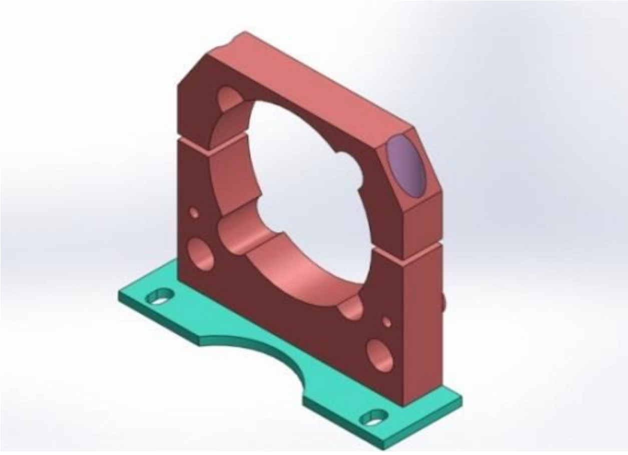


Fig. 4 Q-Rod Mount

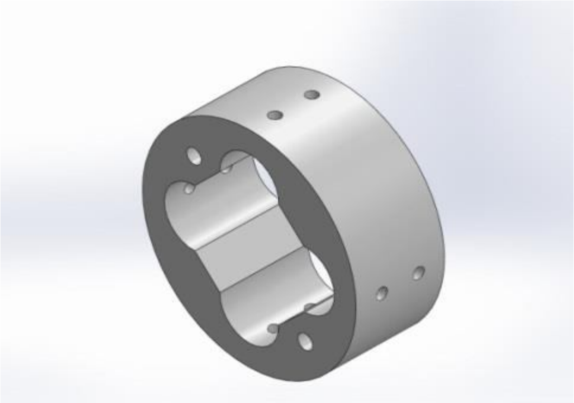


Fig. 5 Ceramic Mount

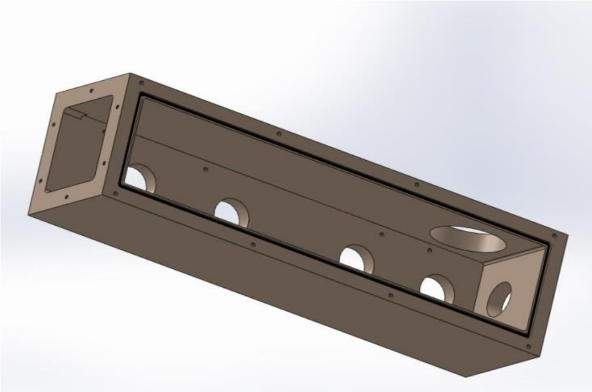


Fig. 6 Outer Chamber

V. FABRICATION DETAILS OF MAJOR COMPONENTS

Fabrication of components plays a vital role in achieving desired accuracy and precision of QMS. Standards such as ASTM 312, ASTM A240, ASTM 213 etc., are used as the basic standards for selecting the materials for the fabrication of the components. Aluminium and stainless steel are selected as the materials for fabrication of the components. SS-316 is the most suitable material for the fabrication of components, but the cost of the material is high. So SS-304 is selected for the fabrication of almost all individual components of QMS. Ion optic system is fabricated using six numbers of circular disc's and fired ceramic spacers, that are needed to be placed between the circular plates for insulation purpose. All the six discs are fabricated from SS-304 sheets, selected according to the ASME code. EDM wire cut, drilling and boring is carried out for fabrication. The filament pins are made up of SS-304 and the insulation is given by a 3mm thick plate of ceramic (fired alumina). The fabrication of filament pins is carried out in CNC spinner lathe. Source insulator is fabricated from Teflon. Nearest dimension is selected and turning operation is done in CNC lathe machine. Source mount and Q-Rod mount are fabricated from SS-304 sheets and plates. Sheets are for higher thickness part and plates for lesser thickness part and are joined together by TIG welding. Thick walled tubes are used as the Q-Rods in order to reduce the weight and to avoid sagging after assembly in the Q-Rod mount. Machining are done in tool room CNC to achieve the required accuracy. For outer chamber, aluminium is used as the material. Conventional machines and CNC machines are used in work shop. Lathe, milling machine, drilling machine, jig-boring machine, EDM wire cut machines etc. are used for fabrication.

VI. ASSEMBLY OF QMS

The better performance of the QMS depends upon the accuracy and alignment of each of the component. Factors such as misalignment of rods must detract from the resolution; hence special care must be taken while assembling. Assembly section of the entire QMS can be divided into three sub-assembly sections that is the assembly of Ionization chamber, analyser, and collector. After the subassembly of the three systems, each subassembly is to be mounted on the vacuum chamber.

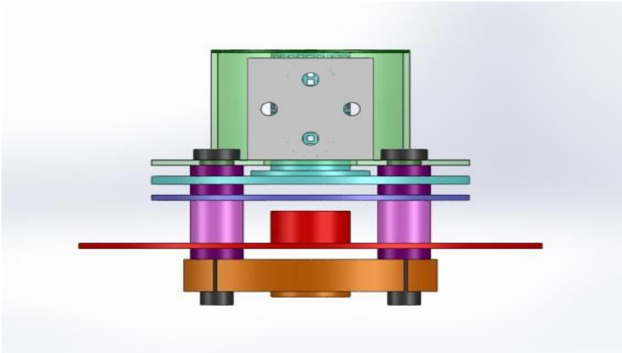


Fig. 7 Ion Optics Assembly

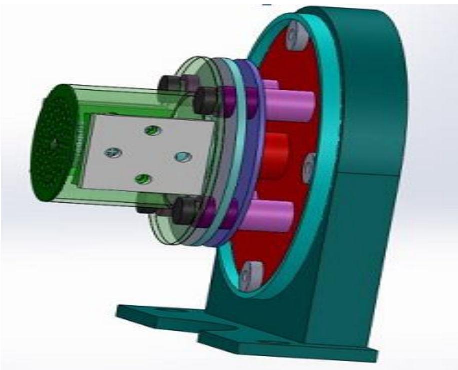


Fig. 8 Ionization Side Assembly

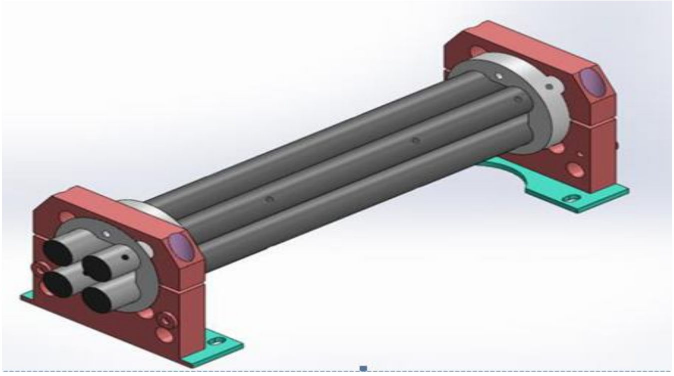


Fig. 9 Analyser Assembly

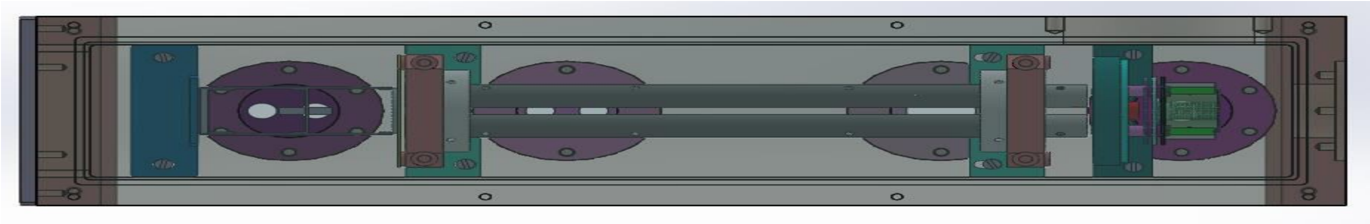


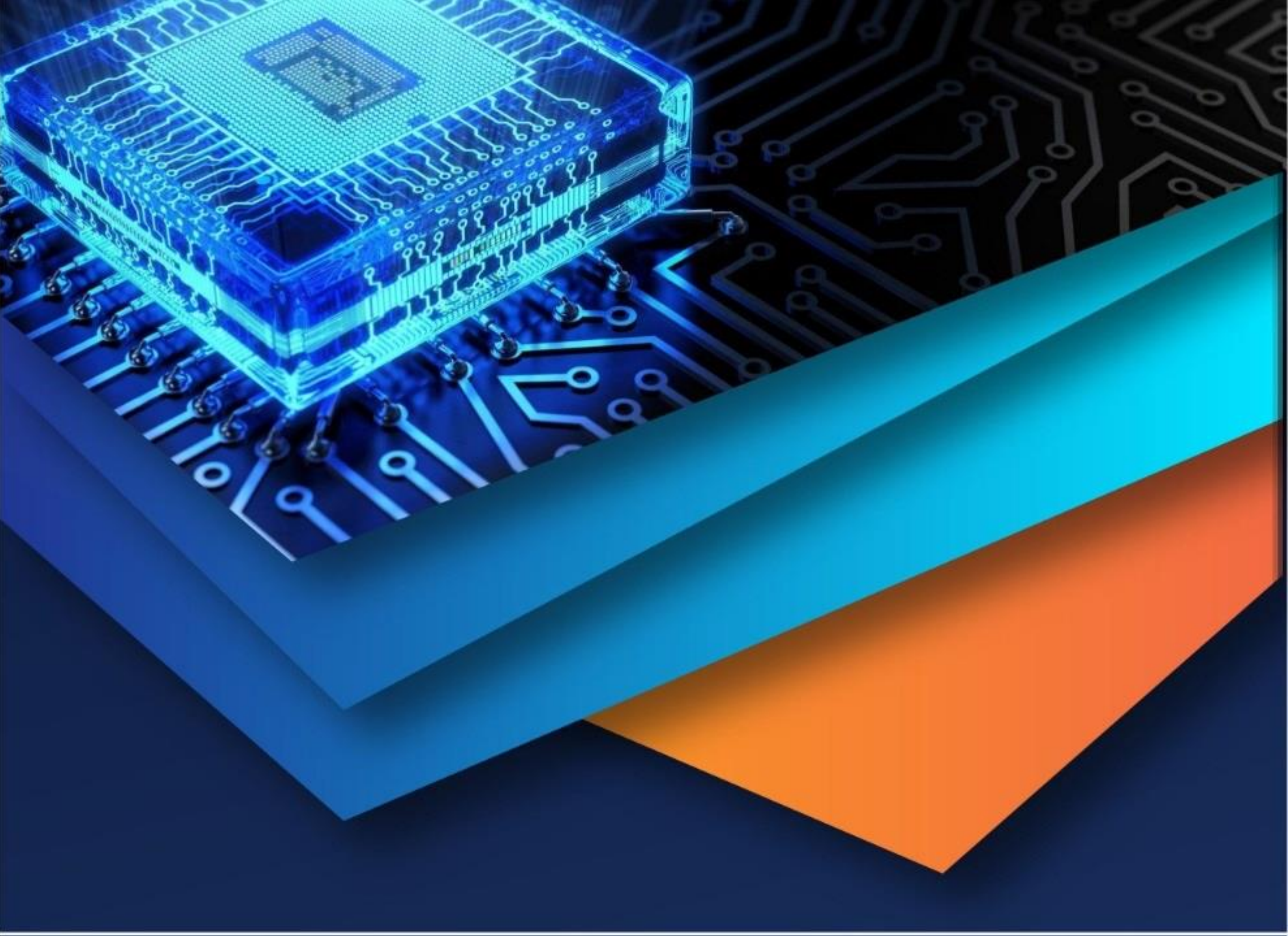
Fig. 10 Entire QMS Assembly (Top View)

VII.CONCLUSION AND FUTURE SCOPE

The Quadrupole mass spectrometer is a important tool to investigate various analysis and highly useful to nuclear field among other fields. It can be even used for a rough idea of isotopic analysis or impurity analysis. Studies have been carried out on ion source, analyser, collector system and the vacuum chamber to improve overall performance of system. Ion optics was re-designed for better transmission and higher electrical insulation for the application of higher potential compared to earlier conventional old design. New filament assembly with ceramic base ensures stable signal for long duration. The vacuum chamber is modified by replacing the circular SS chamber with square aluminium chamber. Aluminium is a better material for vacuum because of its less out gassing. There is scope for further improvements that may form part of further studies. The design of a more open ion source can be worked out for better efficiency of ionization and it require detailed simulated study of the ion generation and propagation. Even though the system has become very compact in the new design, still there is a little scope for further miniaturization by using better packaging arrangement especially in the electronic side.

REFERENCES

[1] Xinyu Si, Lili Hu, Wei Xu, Hang Li, “A dual-source miniature mass spectrometer with improved sensitivity”, International Journal for Mass Spectrometry, 2017, 25, 36-41.
[2] S. K. Aggarwal, H. C. Jain, “Introduction to mass spectrometry”, Indian Society for Mass Spectrometry, 1997,13, 3-8.
[3] S. K. Aggarwal, D. Alamelu, “International Journal of Mass Spectrometry”,2005, 241, 83-88.
[4] R.K. Bhatia, V. K. Yadav, Milind M Gulhane, E Ravisankar, T K Saha, V Nataraju, S.K. Gupta, Meera Murali, M. M. Vilas, M. Gopalakrishna, Mahadevi Sharma, K D Joshi , “Proceedings of ISMAS symposium”,2013,11,19-23.
[5] S. N. Bindal, R K Bhatia, E Ravisankar, V K Handu, “Proceedings of ISMAS symposium”, 2006,10, 55-157.
[6] M. S. Story “Journal of Vacuum Science Technology”,1997, 4, 326-350.
[7] Boris Brkic, Neil France, Adam T. Clare, Chris J. Sutcliffe, Paul R. Chalker and Stephen Taylor, “Development of quadrupole mass spectrometers using rapid prototyping technology”, American Society for Mass Spectrometry,2009,20,1359-1365.
[8] J. Warmenhoven, J. Demarche, V. Palitsin, K.J Kirkby, R.P. Webb , “Modeling transport of secondary ion fragments into a mass spectrometer”, Conference on Application of Accelerators in Research and Industry,2009, 23,352-360.
[9] Raymond E. March, “An introduction to quadrupole ion trap mass spectrometry”, Journal of Mass Spectrometry,1997, 32, 351-369.
[10] V.V.K. Rama Rao, “International Journal of Mass Spectrometry and Ion Physics”, 1983, 52, 289-298.
[11] Michael E Wieser, Johannes B Sehwieters, “International Journal of Mass Spectrometry”, 2005,242, 97- 115. 68
[12] Robert L. Fitzgerald, carol L. O’Neal, “Comparison of an ion trap and a quadrupole mass spectrometer using diazepam as a model compound”, Journal of Analytical Toxicology,1997, 21
[13] P. G. Abichandani, S. S. Misra, S.K. Sahu, K.R. Babu , “Development of quadrupole DC and ion optics”, Indian Society for Mass spectrometry,2014,28, 166-169.
[14] M.M. Gulhane, K.D Joshi, M. Murali, Raju K and T.K Saha, “Improved data acquisition system for QMS”, Indian Society for Mass spectrometry,2017, 31, 165-167
[15] B.R. Gonde, A.M. Kasbekar, M.P. Sharma, M.M. Gulhane, Rabi Datta, T.K. Saha, P. Abhichandani, E. Ravisankar and V. Nataraju, “QMS based hydrogen-steam concentration measurement system”, Indian Society for Mass spectrometry, 2017, 31, 192-194.
[16] Andries P. Bruins ,Journal of Chromatography, 1998,A 794, 345-357.
[17] J. M. Goodings, J. M. Jones and D. A. Parker, “International Journal for Mass Spectrometry and Ion Physics”,1992, 9, 417.
[18] H.J. Heinen, “International Journal for Mass Spectrometry and Ion Physics”,1974, 13,55
[19] H.L. Brown and M.Anbar ,International Journal of Radiation Physics,1975,7,281
[20] A. Benninghoven, C. Plog and N.Treitz , “International Journal for Mass Spectrometry and Ion Physics”, 1996,13,415
[21] D.F Hunt, G.C Stafford and J.W Russell , “Analytical Chemistry”, 1996,48, 2098
[22] R. K. Bhatia, V. K. Yadav, Rabi Datta, M. M. Gulhane, A. M. Kasbekar, T. K. Saha E. Ravisankar, V. Nataraju, S. C. Gadkari, “MS leak detector for UHV applications”, Indian Society for Mass spectrometry,2017,31, 195-197.
[23] W.M. Brubaker, “Advances in mass spectrometry”, Elsevier scientific publishing company, Amsterdam (Netherland),1968
[24] I.T. Platzner, “Modern isotope ratio mass spectrometry”, John Wiley & Sons Ltd.1997
[25] Peter H Dawson, “Quadrupole mass spectrometer and its application”, Elsevier scientific publishing company, Amsterdam (Netherland),2013



10.22214/IJRASET

INDEX COPERNICUS 45.98

ISRA JIF IMPACT FACTOR: 7.129

TOGETHER WE REACH THE GOAL IMPACT FACTOR: 7.429

ISI

INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY