

A Compact Quadrupole Ion Filter for Helium Detection

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A compact quadrupole ion filter was conceived and constructed for optimum performance at the mass four region of the mass spectra. It was primarily designed for geological applications in the measurements of helium of soil-gases. The whole ion filter structure is 15 cm long by 3.5 cm diameter, including ion source and collecting plate. The sensitivity to helium is of the order of 10^{-2} A.torr⁻¹ measured at a total pressure of 6×10^{-6} torr and resolution 6. The system can be easily adapted to work as a dynamic residual gas analyser for other purposes.

Um filtro de íons quadrupolar foi projetado e construído para desempenho ótimo na região de massa quatro do espectro de massas. Esse instrumento foi inicialmente planejado para aplicações geológicas na medida do conteúdo de hélio da fase gasosa dos solos. A estrutura básica do filtro mede 15 cm de comprimento por 3.5 cm de diâmetro, incluindo a fonte dos íons e a placa coletora. A sensibilidade para o hélio é da ordem de 10^{-2} A.torr⁻¹, medida a uma pressão total de 6×10^{-6} torr e resolução 6. O instrumento pode ser facilmente adaptado para operar como analisador dinâmico de gases residuais em outras aplicações.

1. INTRODUCTION

A quadrupole mass spectrometer (QMS) is a dynamic mass analyser used particularly in residual gas analyses. In such analysers, the ion separation is based on the time dependence of one of the system's parameters. There are three types of mass analysers that utilize electrodynamic quadrupolar fields: the quadrupole mass filter¹, the monopole², and the ion trap³. The first one is described in the present work.

The main advantage of the quadrupole mass spectrometer in either of its three versions is the high sensitivity offered in a compact lightweight instrument. VonZahn and Gross⁴, for example, described the utilization of QMS in balloons and light rockets for ionospheric measurements, and Brubaker⁵ has utilized a minute QMS inside the helmet of astronauts for breath analyses.

The primary purpose for the development on the present quadrupole mass filter is to measure the helium content of soil-gas for geological purposes and uranium prospecting^{6,7,8}. However, the same instrument can be easily adapted to be utilized in the high vacuum technology to selectively measure residual gases, simply by modifying the associated electronics.

2. THEORETICAL CONSIDERATIONS

The schematic of the quadrupole mass filter is shown in Figure 1. It ideally consists of a set of two parallel pairs of hyperbolic electrodes subjected to a symmetrical radiofrequency voltage, such that in each of two opposite electrodes the voltage is always of the same magnitude and phase. Superimposed is a direct current bias applied to each opposite pair of electrodes. The resulting electrical potential has the form:

$$V = V_{ac} \cos 2\pi ft \pm V_{dc} \quad (1)$$

where f is the frequency of the alternate field (sec^{-1}), t is the time (sec), V_{ac} and V_{dc} are the applied electrical potentials (volt).

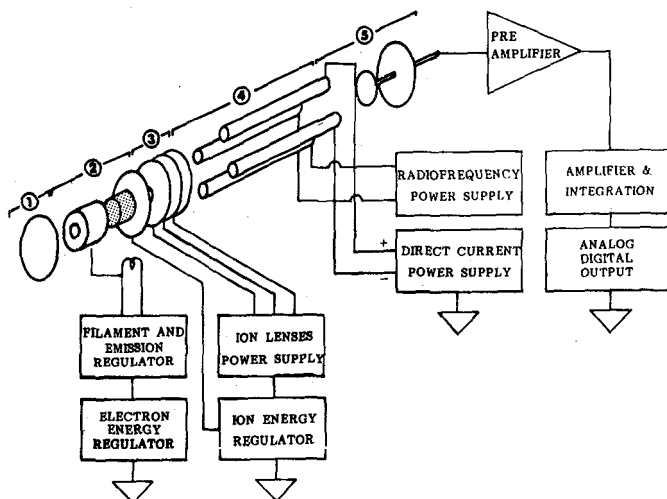


Fig. 1 - Schematic of the quadrupole mass filter.

The ions formed in an electron impact ion source are focused on to the axis of the electrode assemblage where they are subjected to the selective alternating hyperbolic electrical field. Only those ions with a given charge to mass ratio will have a stable path along the quadrupole axis and reach the ion collector where they can be counted. All others ions will be collected by the electrodes and case and neutralized.

The transversal motions of the ions in a hyperbolic field generated by a quadrupole having hyperbolic electrodes are theoretically described in detail by Paul et al.⁹. The bounded solutions for the Mathieu's equations described in the above reference were revised by Arnold¹⁰, Denison¹¹, and Austin et al.¹² and presented in a more workable form in order to obtain simple direct relations between the resolution $m/\Delta m$, the frequency f (sec^{-1}), the electrical potential V_{ac} and V_{dc} (volt), the electric driving power P (watt), and the electrode clearance r_0 (meter) for all ions having the same charges to mass ratio:

$$V_{ac} = 7.14 \times 10^{-8} f^2 r_0^2 m \quad (2)$$

$$\frac{V_{dc}}{V_{ac}} = 0.1678 - 0.126 (\Delta m/m) \quad (3)$$

$$V_z = \frac{L^2 V_{ac}}{570 r_0^2} (\Delta m/m) \quad (4)$$

$$P = 6.5 \times 10^{-26} C m^2 f^5 r_0^4 / Q \quad (5)$$

where m is the ion mass (amu), L is the length of the electrode assemblage (meter), V_z is the ion potential (volt), and C , Q are the net capacitance (pF) and circuit quality factor, respectively.

The above equations are only valid for perfectly hyperbolic electrode rods. However, the highly sophisticated machinery necessary to construct and adjust hyperbolic electrodes leads most quadrupole mass filters to use circular electrodes instead. Circular electrodes have the ideal hyperbolic electrical field only near the axis of the rod assemblage. Denison¹¹ theoretically shows that a circular electrode clearance $r_c = 1.1468 r$, where r is the radius of the circular electrode, have the best possible approximation to the hyperbolic electrode assemblage. The importance of the proper choice of r_c is more pronounced in the power requirements needed to drive the mass filter, since r_0 is raised to the fourth power in equation (5).

Table 1 shows the electrical parameters for the present version of the quadrupole ion filter in its entire mass range, calculated from the above formulas. Although V_z can be as low as a fraction of volt for the hydrogen range of mass, there is actually a lower experimental limit for the ion energy in order to allow ions be efficiently injected and transmitted in the hyperbolic field (some 3 volt for the present instrument). This is due to the effect of fringe electrical fields at the ends of the quadrupole electrodes, which greatly diverges from the ideal hyperbolic electrical field. The shorter the time an ion spends in this region (the higher V_z), the less it deviates from the axis of the electrode assemblage, and the better is the ion transmission (see, for example, Brubaker¹² for ingenious solution for this problem).

Table 1. Electrical parameters (volt) for the quadrupole ion filter (*) in the entire mass range of the instrument.

$m(\text{AMU})$	$m/\Delta m$	3	6	9	12	15	18
1 (H^+)	V_{ac}	5.88	5.88	5.88	5.88	5.88	5.88
	V_{de}	0.74	0.86	0.90	0.92	0.94	0.94
	V_z	*.48	2.24	1.49	1.12	0.90	0.75
2 (H_2^{++})	V_{ac}	11.70	11.70	11.70	11.70	11.70	11.70
	V_{de}	1.48	1.72	1.81	1.85	1.90	1.91
	V_z	8.96	4.48	2.97	2.3	1.79	1.49
3 (He^+)	V_{ac}	17.64	17.64	17.64	17.64	17.64	17.64
	V_{de}	2.22	2.59	2.71	2.77	2.81	2.84
	V_z	13.44	6.72	4.48	3.36	2.69	2.24
4 (He^+)	V_{ac}	23.54	23.54	23.54	23.54	23.54	23.54
	V_{de}	2.96	3.46	3.62	3.70	3.75	3.78
	V_z	17.94	8.96	5.98	4.48	3.58	2.99
8 (O^{++}) **	V_{ac}	47.08	47.08	47.08	47.08	47.08	47.08
	V_{de}	5.92	6.91	7.24	7.40	7.50	7.56
	V_z	35.89	17.94	11.96	8.97	7.17	5.97

* $f = 3.2768$ MHz, $r_0 = 0.277$ cm, and $L = 10$ cm.

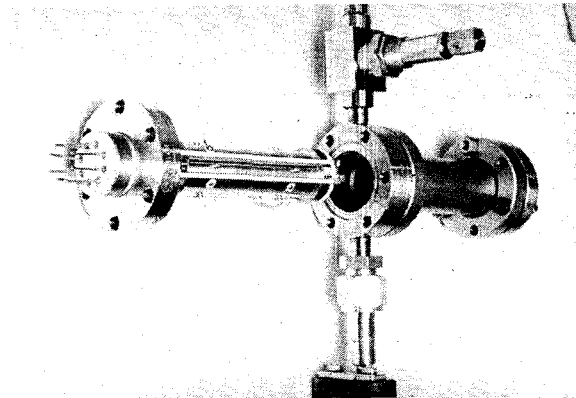
** Out of the mass range of this instrument with the present electronics.

3. DESCRIPTION OF THE INSTRUMENT

The mass filter described here was primarily designed to be small and lightweight for easy field transportation in geological applications. Furthermore, it was designed to have maximum performance in the mass-4 region in order to measure natural helium always present in variable concentrations in the soil-gas. For this reason, this instrument has a limited mass range in order to save some weight and power requirements in the peripheral electronics.

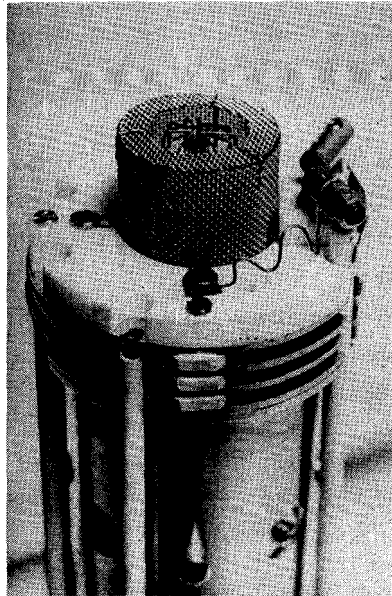
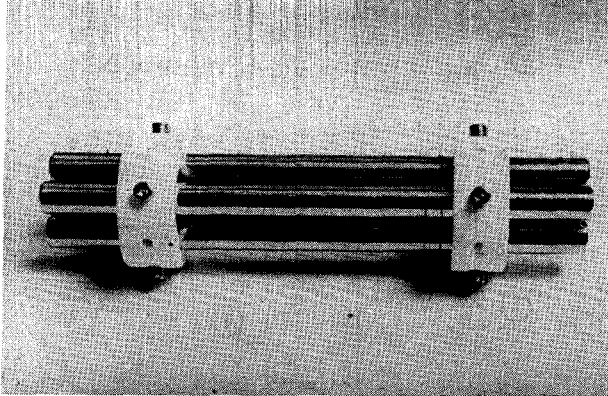
The complete mass filter is 15 cm long and 3.5 cm diameter (Picture 1) and fits inside a standard stainless steel vacuum case^(*) with a rough vacuum valve and an 8 l/sec vacuum pump attached to one end. An electrical feedthrough provides both the mechanical support to the mass filter as well as the electrical connections between the vacuum side of the instrument and the external electronics. The circular electrode assembly is made out of non-magnetic stainless steel rods and is mounted on a set of two glass-ceramic^(**) insulators. The assemblage is shown in Picture 2.

The ion source is a grid-type low gas desorption (Hofman¹³), using a circular 0.001" thick tungsten filament (Picture 3). An ion focusing set of three cylindrical lenses follows the ions source. Figure



* Varian, nipple 952-5048, flange 952-5001, feedthrough 954-5014.

** MACOR, Corning Glass works.



2 schematically illustrates the ion source and focusing set. Lenses L_1 , L_2 in conjunction with lens L_3 form an accelerate-descelerate arrangement that provides a net focusing effect to the positive ion beam. The net ion energy is essentially not affected by the focusing lenses assembly and is given by the potential difference between source 7 and the ion collecting plat at ground potential (not shown in the scheme).

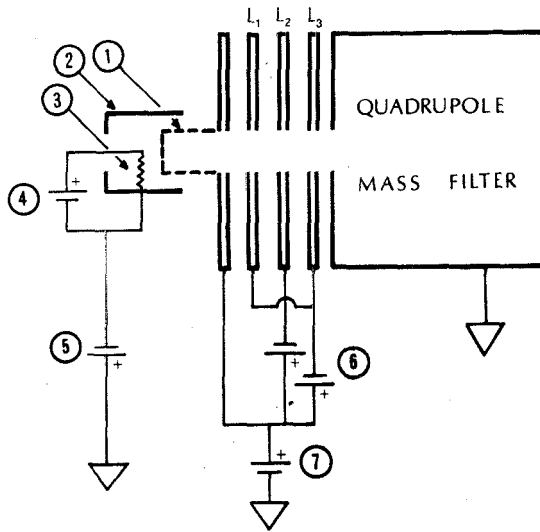


Fig. 2 - Schematic of the ion source and ion focusing set 1) screen; 2) electron repeller; 3) filament; 4) filament power supply (up to 1200 mA); 5) electron voltage power supply (-20 to -100 V); 6) ion lenses power supply (+20 to -120 V); 7) ion energy (+5 to +20 V).

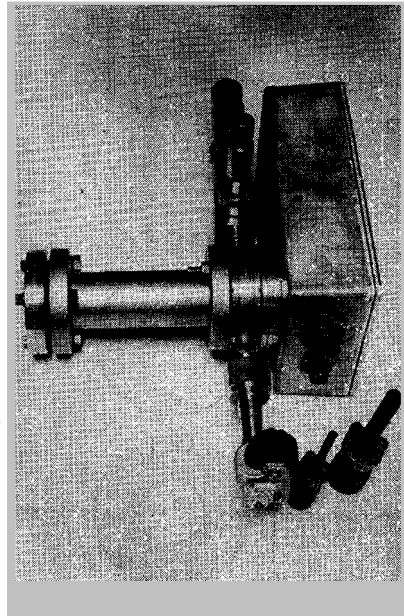
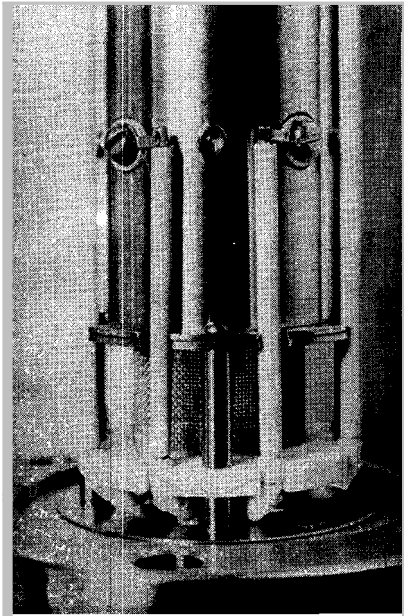
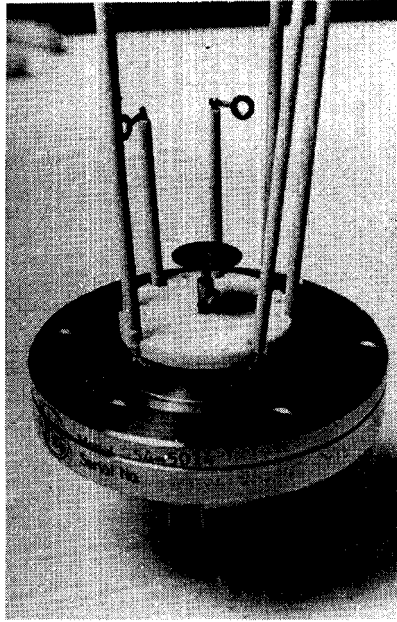
The ions are collected by a single shielded plate (Pictures 4 and 5), after passing through the ion filter. In order to minimize noise capture, the connection of the ion plate with the external electrometer amplifier is kept as short and stiff as possible. The solid state amplifier^(*) is assembled inside a solid cast aluminium box directly attached to the quadrupole case (Picture 6). This aluminium box also provides all electric outlets for the ion filter.

Exact dimensions and engineering details of the quadrupole ion filter, ion source, and collector are given in Appendix 1.

4. OPERATING CHARACTERISTICS

The neutral atoms are introduced in the source at pressures lower than 10^{-4} torr (typical pressure is 10^{-6} torr). Ions are generated by the impact of electrons produced in a hot tungsten filament inside

* Analog Devices, model 310J.



the ion source. Optimum filament current for the present instrument is within 900 to 100 mA. Larger currents will increase the ionization rate but will also cause overheating of the case and decrease the lifetime of the filament. The filament temperature is current regulated such that the emission current is always constant to a pre-set value, by using an error amplifier that feeds-back the filament source circuit. Typical electron energies for the present instrument are between 20 to 100 electron-volt and are chosen in order to optimize the production of a given ion while keeping molecular fractionation to a minimum (lower noise). For the case of the helium-4, this energy was found to be about 80 electron-volt.

The positive ions are extracted from the ion source by a gentle electrical potential of approximately -50 volt applied to lens L_1 , measured with respect to the ion potential reference (see Figure 2, source 7). Lens L_2 is at the same potential as L_1 , and the potential at L_3 is chosen such that there is a net accelerate-descelerate effect to the ion beam. The accelerate-descelerate arrangement of lenses was preferred to the descelerate-accelerate arrangement because it keeps the ion beam closer to the axis where perturbations due to mechanical imperfections are fairly unimportant (see, for example, Septier¹⁴). The optimum transmission of ions in the lower mass range (4 amu) was found when potential at L_3 was about -100 volts.

The ion energy was adjusted in a semi-empirical way, partially based on the theoretical results of Table 1 for V_z and also by monitoring the output of the system for maximum ion transmission at mass four. Results thus obtained for V_z was observed to be slightly higher than theoretically predicted values (about 3 volt higher).

A crystal controlled radio frequency power supply operating at 3.2768 MHz with a V_{ac} maximum of 50 volt furnished the necessary driving power to the quadrupole electrodes. The output transformer of the radio frequency supply is center taped to ground for a symmetrical output. The necessary symmetrical D.C. bias is obtained by properly rectifying and filtering the R.F. outputs. This procedure allows a fairly constant D.C. to R.F. ratio which is a primary requisite for a good spectral resolution.

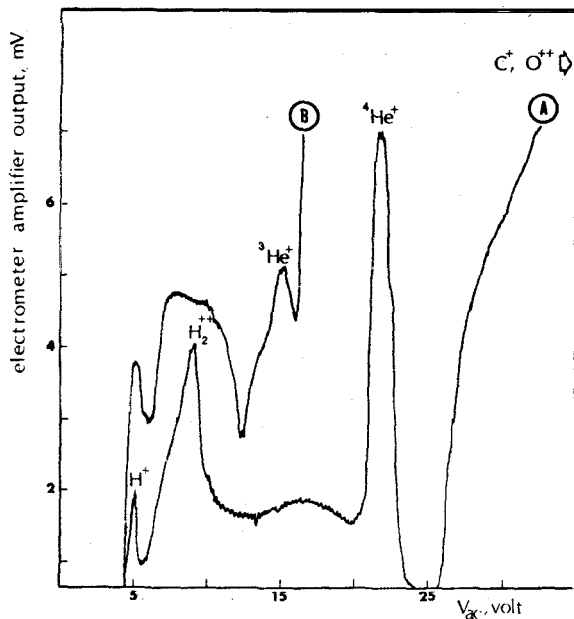


Fig. 3 - Typical mass spectra in the mass four region.

Figure 3 shows typical mass spectra obtained for the helium-4 mass region. The spectra were obtained in a dynamic mode at 6×10^{-6} mmHg internal pressure. The spectrum A is at resolution 6 and spectrum B is at resolution 2.5 calculated at the helium-4 peak from equation (3). The effect of broadening of the peaks and increased sensibility at lower resolutions is immediately observable. Sensitivity to helium ions for the whole instrument was nearly 10^{-2} A.torr $^{-1}$, measured using the above configuration.

The ion lenses were observed to be discriminative to a certain extent. This is seen in Figure 4 obtained with similar conditions of the spectrum A in Figure 3 but with L₁ potential decreased to about -250 volt. With this lens arrangement all peaks of masses lighter than helium-4 were eliminated from the spectrum.

The stability of the instrument is essentially dependent on the stability of the associated electronics. Nevertheless, some random gain drifts were observed to be related to the lack of mechanical stiffness in the connection between the electrometer amplifier and the ion collecting plate.

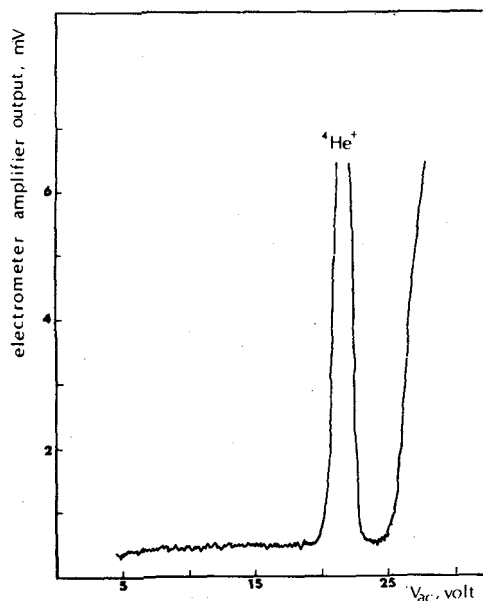


Fig. 4 - Mass spectrum showing the effect of mass discrimination of the ion lenses.

5. CONCLWSIONS

Preliminary tests¹⁷ with the instrument using artificially prepared helium samples have demonstrated that it has enough sensitivity and stability to be used as helium detector for geological porpouses. Nevertheless, it is still necessary to develop an efficientsample inlet for this specific purpose.

Although the quadrupole ion filter described here was optimized for the helium region of masses, it is the opinion of the present authors that it can also be used in an extended mass range with only a few modifications in the associated electronics. Basically, the R.F. maximum amplitude shall be augmented following values given by equation (2). The D.C. to R.F. ratio will be automatically kept constant if it is being used the electronic configuration suggested in the text. The ion energy shall be adjusted following values given by equation (4).

The described QMS in its extended mass configuration can probably be used as a compact and sensitive dynamic residual gas analyser in

the ultra high vacuum technology, pollution, and air control sciences, medicine, and high atmosphere research.

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