

# The physics and technology of quadrupole mass spectrometers



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## ABSTRACT

The fundamental principles of quadrupole mass spectrometers (QMS) are discussed, concentrating on instruments used in vacuum applications.

The mass-selective element is the quadrupole mass filter, first described in a 1956 patent. Although originally intended for isotope separation, the concept soon became applied to analytical chemistry and to residual gas analysis (RGA), also known as partial pressure analysis (PPA). The ion optical design of a typical general-purpose quadrupole RGA has altered little since the 1970s – although of course there have been major advances in electronics packaging, data capture, and automation. There is growing interest in miniaturised systems for high-pressure work.

These instruments mostly use electron-impact ionization sources, based on principles established nearly 100 years ago. Those used for vacuum applications are generally of two types: an “open” design, rather like a miniature Bayard-Alpert gauge, and a “closed” design, generally based on Nier’s work.

The simplest and most robust detector is a Faraday collector; alternatively an electron multiplier is used when the highest sensitivity is required.

Often the quadrupole drive electronics and the amplifier are mounted directly on the vacuum flange, giving a compact assembly. Instrument control and data acquisition is almost always done via a computer interface. This gives a very versatile and powerful system. Nevertheless, to obtain the maximum benefit from any mass spectrometer it is often necessary to keep in mind the underlying physical principles.

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## 1. Introduction

The concepts of mass spectrometry can be traced back to work by Aston at Cambridge and by Dempster at Chicago. Dempster’s 1918 paper [1] clearly shows the vacuum system, ion source, mass analyser and detector – all the basic functionality needed for a mass spectrometer. Dempster used the instrument to study isotope distributions of volatile metals (Mg, Li, K, Ca, Zn).

In a quadrupole mass spectrometer (QMS) an alternating electric field generates the mass-selective action. The small size of the quadrupole, its relatively low cost, modest power requirements and linear mass scale have all contributed to its widespread acceptance as an instrument for residual gas analysis (RGA) in vacuum systems [2–4].

The quadrupole was first described by Paul & Steinwedel [5], see also Paul & Raether [6]. Although we know the quadrupole primarily as an analytical instrument, it was originally conceived with isotope separation in mind. Finlan et al. [7] describe an

experimental system built at Amersham in which the rods were 3 m long with an  $r_0$  (inscribed radius) of 1.35 cm. They report direct observation of the ion beam in the quadrupole, but unfortunately there are no photographs. Von Zahn [8] describes a system that used tensioned molybdenum wires to define the electrode surfaces: the electrodes were 5.82 m long with a hyperbolic profile. The inscribed radius was 3.5 cm. The total tension from the multiplicity of wires amounted to nearly 1000 tons.

Of course most quadrupole instruments are much more compact. Instruments for general-purpose research or for chemical analysis, such as gas chromatography mass spectrometry (GC–MS) or inductively coupled plasma mass spectrometry (ICP–MS) are usually 200 mm to 300 mm in length, with an inscribed radius of 6 mm–7 mm. A mass filter for residual gas analysis (RGA) is typically 100 mm to 150 mm in length with an inscribed radius of around 3.5 mm. It is usually built on a standard UHV-compatible 70 mm flange with electrical connections made via a multi-way feed-through. The drive electronics will usually be very compact, and mount directly onto the vacuum flange. The basic mechanical design of the mass filter and of RGAs in particular has altered little. Much of the commercial development during the past thirty years or so has concentrated on the electronics and data acquisition.

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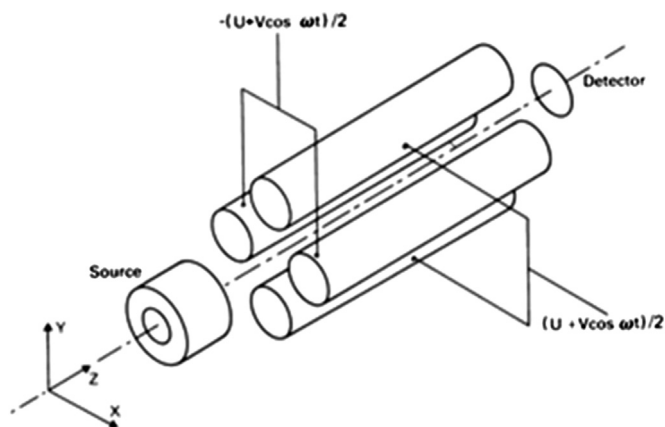


Fig. 1. Quadrupole mass spectrometer for residual gas analysis.

When the author started working with RGA quadrupoles, a data system was something of a novelty. Today it would be inconceivable to manufacture an instrument without one!

Much smaller RGAs have been developed recently [9]. This is largely driven by the desire to operate at relatively high pressure, since small dimensions make for reduced losses by ion/molecule collisions and reactions.

## 2. The quadrupole mass spectrometer (QMS)

The main functional components of any mass spectrometer are in principle the same as in [1], although usually we would add an inlet system, electronics and a data system for completeness. The main components of a QMS for RGA are shown in Fig. 1.

### 2.1. Inlet system

Often the RGA is installed in the environment it is to monitor, so there is no inlet system as such. But in coarse vacuum, or for calibration, we may need to install the RGA in a separate differentially pumped environment, so that it can operate at a manageable pressure. If so, we must ensure that the gas composition within the ion source is representative of the composition to be monitored. Therefore the inlet system should be designed so as to avoid fractionation [10].

When the sample is at a pressure higher than the operating pressure for the RGA itself (typically  $10^{-6}$  to  $10^{-5}$  mbar) the sample has to be introduced via a pumping restriction. This might be a valve or a simple aperture as indicated in Fig. 2a. It can be advantageous to operate the ionization volume at a pressure (typically  $10^{-4}$  mbar) that is higher than the pressure in the main RGA housing. Such a “differentially-pumped” source is common in analytical work, see Fig. 2b. The filament is outside the ionization

volume and so operates at a reduced pressure. This is beneficial for filament lifetime and reduces unwanted interaction between the filament and the sample which can cause problems if reactive gas species are to be analysed [11].

### 2.2. Ion source

There are many different types of ion source, but for general-purpose analytical work, electron-impact ionization [12] is usually employed. The ion source may be quite complex – it may incorporate magnets to confine the electron beam [13], and perhaps selectable aperture sizes to change the pressure within the source. But for RGA a simpler arrangement, based on a design [14] intended originally for molecular beam work, usually suffices.

A typical RGA source is shown schematically in Fig. 3. It resembles a small extractor gauge. Electrons are emitted from an electrically-heated filament. It is made from, or coated with, material with a relatively low work function, so that electrons are readily emitted when it is heated.

The electrons are accelerated towards and through a grid to enter the ionization region with enough energy (typically 40 eV–100 eV) to ionize the sample. The electron trajectories are complex, and may traverse the ionization region several times before eventually impacting on the source grid. The emission current (that is, the current of ionizing electrons) is monitored at the source electrode, and is regulated by controlling the heating current that flows through the filament. In effect, the filament and the source electrode behave like the cathode and anode of a thermionic diode valve. Ions formed in the ionization volume are extracted by means of an electric field applied to an aperture plate, and are directed into the mass analyser.

The electron energy is essentially determined by the voltage applied between the filament and the source electrode. It is often set to around 70 eV, which is close to the optimum for most gas species. For some applications it is advantageous to set it lower; this reduces fragmentation and the tendency to form multiply-charged ions, and so simplifies the mass spectrum. For example, when analysing trace levels of water in argon, there is an interfering peak at mass 18 from doubly-ionized  $^{36}\text{Ar}$  which is equivalent to around 300 ppm of water. By reducing the electron energy to 40 eV the detection limit of water can be improved by at least a factor of 10.

The sensitivity is proportional to the number of electrons available for ionization, and therefore in principle to the emission current; it is usually expressed in terms of the signal current per unit of pressure. The heating current flowing through the filament is typically 2–3 A and the emission current is typically 1 mA; this can be expected to give a sensitivity of around  $10^{-4}$  A/mbar for a simple gas species such as nitrogen or argon.

A voltage, usually in the range 5–10 V, is applied to the source electrode. The extractor electrode is driven with a negative voltage, typically in the range –50 V to –130 V. This establishes an electric field that penetrates into the ionization region and helps to direct

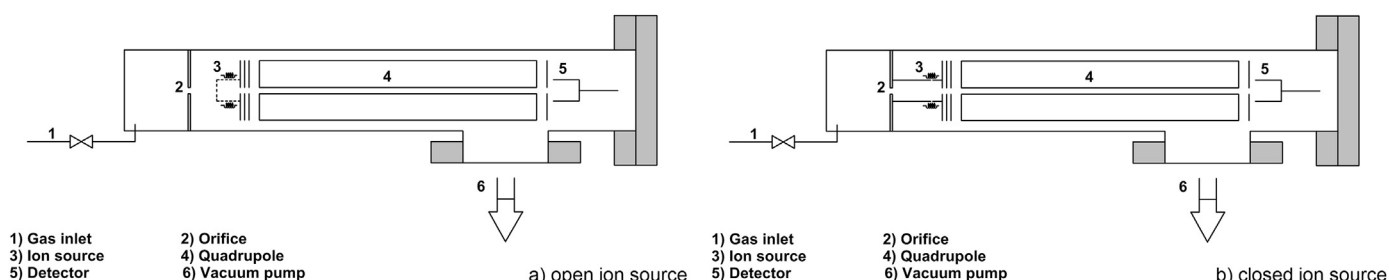


Fig. 2. Sampling from a higher pressure.

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